NASA GR 10649Q

# CRITICAL ANALYSIS OF ACCUMULATED EXPERIMENTAL DATA ON FILAMENT-REINFORCED METAL MATRIX COMPOSITES

John A. Alexander Robert G. Shaver James C. Withers

June 1969



Prepared Under Contract No. NASW-1779 by

GENERAL TECHNOLOGIES CORPORATION 1821 Michael Faraday Drive Reston, Virginia 22070

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

# CRITICAL ANALYSIS OF ACCUMULATED EXPERIMENTAL DATA ON FILAMENT-REINFORCED METAL MATRIX COMPOSITES

John A. Alexander Robert G. Shaver James C. Withers

June 1969

Prepared Under Contract No. NASW-1779 by

GENERAL TECHNOLOGIES CORPORATION 1821 Michael Faraday Drive Reston, Virginia 22070

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

#### **FOREWORD**

The extraordinary potential of composites as a new type of material which can be tailored in properties to meet the requirements of a wide variety of high performance structural applications has led to heightened recent interest and a substantial volume of research activity in the area of continuous fiber reinforced metals. The rapid expansion of experimental effort has created a large body of data which requires critical analysis and review. The accelerated development of this particular type of composite into a practical material for aerospace spplication has made difficult the task of assimilation by individual researchers of the total volume of data being generated from the various active organizations.

This critical analysis and review of the research and development accomplishments in the metal matrix composites field is intended to consolidate the observations of the work conducted since the Kelly and Davies<sup>(1)</sup> and Cratchley<sup>(2)</sup> papers of 1965 in Metallurgical Reviews, cross-correlate the data, and compare the results and conclusions for internal consistency and compliance with the theoretical predictions for composite behavior. The ultimate objective of this work is to indicate research and development areas where focused activity can contribute most to the provision of metal matrix composite materials to satisfy critical NASA needs.

The review and analysis covers continuous fiber reinforced metal-matrix composites, their fabrication, properties, and problems.

#### **ABSTRACT**

A critical review is presented of the experimental accomplishments in the field of filament reinforced metal matrix composites. The review covers the experimental work reported in the period 1965 through 1968. It treats the fabrication processes which have been employed to produce a variety of composite forms in terms of the processing parameters which determine ultimate properties. The mechanical property data have been treated in a manner which emphasizes the character of metal matrix composite performance rather than the absolute values of the specimens tested. Finally the review underlines the residual problem areas and suggests experimental approaches to the advancement of composite technology.

### TABLE OF CONTENTS

			Page
i	INTR	ODUCTION	1
11	SUM	MARY	3
Ш	FABR	ICATION PROCESS DEVELOPMENT	10
	1.	HOT-PRESSURE BONDING	10
	2.	LIQUID METAL INFILTRATION	12
	3.	ELECTRODEPOSITION	15
	4.	PLASMA SPRAYING	24
	5.	CHEMICAL VAPOR DEPOSITION	28
	6.	COLD PRESS AND SINTERING	30
	7.	EXTRUSION AND ROLLING	30
	8.	HIGH ENERGY RATE FORMING	31
IV		MECHANICAL PROPERTIES OF FILAMENT REINFORCED AL MATRIX COMPOSITES	36
	1.	TENSILE PROPERTIES AT ROOM TEMPERATURE	36
		<ul> <li>a. Tensile Modulus</li> <li>b. Tensile Strength</li> <li>c. Filament Orientation Effects upon Composite</li> </ul>	41 45
		Tensile Properties d. Strain Rate Effects Upon Ultimate Tensile Strength and Modulus	45 49
		e. Notch Sensitivity f. Poisson's Ratio	61 61
	2.	ELEVATED TEMPERATURE PROPERTIES OF COMPOSITES	64
		<ul> <li>a. Short-Time Elevated Temperature Tensile Properties</li> <li>b. Elevated Temperature Elastic Modulus</li> <li>c. Creep and Stress Rupture</li> <li>d. Thermal Shock or Cycling Effects</li> </ul>	64 68 68 81
		e. Elevated Temperature Exposure	8

## TABLE OF CONTENTS (cont.)

			Page
	3.	DYNAMIC PROPERTIES	85
		<ul><li>a. Fatigue</li><li>b. Damping Capacity</li><li>c. Impact Properties</li></ul>	85 97 97
	4.	MISCELLANEOUS PROPERTIES	97
		<ul> <li>a. Compressive Properties</li> <li>b. Flexure Strength</li> <li>c. Torsional Properties</li> <li>d. Shear Properties</li> <li>e. Corrosion Properties</li> <li>f. Joining Properties</li> </ul>	99 104 106 106 111
٧	PRO	BLEMS	116
	1.	CONSISTENCY	117
	2.	COST	118
	3.	FORM	119
	4.	FILAMENT	120
	5.	BEHAVIOR	120
VI	CON	CONCLUSIONS	
VII	REC	RECOMMENDATIONS	
REFER	ENCES		126

## LIST OF ILLUSTRATIONS

Figure		Page
1	Composite Forms Which Can be Fabricated by Currently Available Processes	7
2	Demonstrated Properties of Advanced Metal Matrix Composites	8
3	Basic Variations on the Hot-Pressure Bonding Process	11
4	Hot-Pressed Composite Panels	13
5	Airfoil Hot-Pressure Forming Schemes	14
6a	Vacuum Infiltration	16
6b	Schematic Representation of Continuous Casting Process	16
7a	Typical Cross Section of a Continuously Cast Rod	17
<i>7</i> b	Cross Section of a Continuously Cast Rod Showing Almost Perfect Hexagonal Packing of Filaments	17
8	Schematic of the Electrodeposition Process for Forming Aligned Filament Composites	18
9	Schematic Representation of Electroformed Composite Formation	20
10	Filament-Reinforced Monolayer Tape	21
11	Cross Section of a Typical Si-Ni Composite, Specimen #27, as Polished, (150X)	22
12	Void Formation in Ni-W Composites	23
13	Motor Case Section of Filament-Wound Boron in Aluminum Matrix	25
14	Plasma Spray Process	27
15	Application of Helical Wrapping of Tungsten Wire to Mandrel	29
16	Schematic of Pneumatic Impaction Machine Used to Consolidate Fiber Reinforced Composites	32
17	Effect of Specimen Length on the Tensile Strength of SiC Filaments	38

Figure		Page
18	Composite and Constituent Stress–Strain with Tensile Residual Stresses in the Matrix	39
19a	Be Wire Reinforced Aluminum	42
19b	Aluminum Matrix Composite Rule of Mixtures Representation of Moduli	43
19c	Modulus Values for Reinforced Titanium Composites	44
20	Tensile Strength of Al Matrix Composites as a Function of Volume Fraction Filament	46
21	Range of 2024 Al Matrix Composite Properties vs Volume Percent Loading	47
22	The Tensile Strength of Uniaxial Cu–W Composites Tested at Various Angles to the Incorporated Filament	48
23	Tensile Strength Results on Laminated Crossply Panels with Filaments Oriented $\pm\theta$ to the Specimen Axis	50
24	Uniaxial Composite Tensile Properties as a Function of the Angle Between the Fiber Axis and the Tensile Axis	51
25	Fractured Surfaces of Borsic-Aluminum Composites. Specimens Were Tested with Given Angle Between Fiber Axis and Tensile Axis	52
26	Tensile Strength Variation with the Angle Between Uniaxially Oriented Filament and the Tensile Axis	53
27	Effect of v/o Filament on Off-Axis Tensile Properties	54
28	Effect of Aluminum Alloy on Off-Axis Tensile Properties of Boron Reinforced Composites	55
29	Effect of Aluminum Alloy Heat Treatment upon the Off-Axis Tensile Properties of Be Reinforced Composites	56
30	Off-Axis Properties of Al-34 v/o Be and Al-50 v/o B Orthogonal Crossply Composites	57

Figure		Page
31	Comparison of Off-Axis Properties of Uniaxial 50 $v/o$ Boron Filament Reinforced Aluminum with 0-90° and $\pm 30^{\circ}$ Crossplys	58
32	Modulus of Elasticity Versus Orientation for 50 v/o Uniaxial, 0-90° Crossply and $\pm 30^\circ$ Crossply Aluminum-Boron Composites	59
33	Modulus of Elasticity Versus Wire Orientation	60
34	Effect of Strain Rate on Ultimate Tensile Strength Borsic- Aluminum Composites (Nominal Fiber Content 50%)	62
35	Effect of Strain Rate on Modulus and Tensile Strength of Al-65 v/o B Composites	63
36	Tensile Strength of Filament Reinforced Aluminum Composites as a Function of Temperature	65
37	The Elevated Temperature Tensile Strength of Tungsten Reinforced Nickel Alloys as Compared to Matrix Properties	66
38	The Effect of Temperature upon the Tensile Strength of A Representative Composite in the AI, Ti and Ni Matrices	67
39	Effect of Temperature on Nickel Matrix and Titanium Composite Modulus of Elasticity	69
40	The Effect of Temperature Upon the Elastic Modulus of Al Matrix Composites	70
41	The Effect of Temperature Upon the Elastic Modulus of Al Matrix Composites	71
42	Creep Properties of AI-SiO <sub>2</sub> Composites and Other Materials at 400°C	73
43	Creep of Hastelloy X Composite and Commercial Grade Hastelloy X	74
44	The Stress Rupture Life of Aluminum Matrix Composites as a Function of Temperature and Reinforcing Filament	76

Figure		Page
45	Comparison of 500°C Stress Rupture Life of Al-50 v/o Borsic Composite with Titanium-6 Al-4V Alloy	77
46	Comparison of the 500°F Stress Rupture Properties of Boron and Stainless Steel Reinforced Composites with the Properties of the Matrix	<i>7</i> 8
47	100 Hour Rupture Stress of Nickel Base Alloy Composites Reinforced with Tungsten Wires as Compared with Fiber-Free Hastelloy X	79
48	Stress Rupture of Hastelloy X Plus 37 v/o Continuous Tungsten or TZM Molybdenum Wires and Fiber-Free Hastelloy X at 1800 F	80
49	Stress Rupture of Hastelloy X Plus 36 v/o Continuous Tungsten Wires and Fiber-Free Hastelloy X at 1900 and 2000 F	82
50	Effect of 800°F Thermal Shock on Tensile Strength of Al-27% B Composites	83
51	Room Temperature Strength of Boron and Borsic Fibers Heated in 2024 Aluminum	84
52	Al-42 v/o Boron Reverse Bending Flexural Fatigue Test Results for 0° Filament Orientation Specimens	86
53	Combined Axial/Flexural Fatigue Results on Al-B Composites (A = $0.95$ )	87
54	Axial Fatigue Results for Al-B Composites	88
55	Direct Stress Fatigue Life of Hastelloy X Composites and Fiber-Free Hastelloy X at Room Temperature	90
56	Fatigue Behavior of Silver and Silver-W Composites	92
57	Comparison of Continuous and Discontinuous W Reinforced Silver Composites in Fatigue	93
58	Semi-plastic-strain-range, calculated from the idealized model versus log cycles to failure for aluminum/silica and aluminum/stainless steel composites with $5 \times 10^{-2}$ mm ( $2 \times 10^{-3}$ in.) diameter fibers	95

Figure		Page
59	Temperature Versus Log Cycles to Failure for Aluminum/Silica Composites tested Various Stresses	96
60	Comparison of Damping Capacity of Al-SiO <sub>2</sub> Composites with Conventional Engineering Materials	98
61	Predicted and Experimental Strength vs Packing Density for B/Mg Composites	100
62a	Accordion End Failure in a Boron–Magnesium Compression Specimen	101
62b	Mushroomed End Failure in a Boron–Magnesium Compression Specimen	101
62c	Longitudinally Delaminated Boron-Magnesium Compression Specimen	102
63	Compression Stress-Strain Curves of Unreinforced and B-Reinforced X7002 Al Alloy	103
64	Strength Versus Temperature for Aluminum-Boron Composites	105
65	Maximum Shearing Stress in Torsion for Al-B Composites	107
66a	Types of Shear Test Specimens	108
66b	The Effect of Temperature on Shear Strength for Unidirectional and Crossply AI-B Composites	109
67	Geometrically Dissimilar Short Shear Beam Specimens	110
68	Average Shear Modulus Plate Test Results Vs Test Temperature for the Various Filament Orientation Al/Boron Panels and 2024–O Aluminum Sheet	112
69	Environmental Test Specimen	113
70	Schematic Drawing of Six Layer Bonded Composite with Seams Transverse to Gage Section	115

### LIST OF TABLES

Table		Page
1	Fabricationally Stable Filament Reinforced Composite System	5
11	Tensile Properties of Electrolytically Deposited Al-B NOL Rings	26
Ш	Types of Data Utilized to Evaluate Fabrication Process Development	34
<b>IV</b>	Poisson's Ratio Values for Various Al-B Composites	61
٧	Comparison of A1-50% BORSIC Composite Short-Time Tensile with 0.1 Hour Stress Rupture Strengths	<i>7</i> 5
VI	Comparison of $\frac{\sigma_{cf}}{\sigma_{c}}$ for Hastelloy Composites and Matrix	
	at Various Temperatures	89
VII	Fatigue "Facts"	91
VIII	Comparison of Tensile and Compressive Strength of Unidirectional Aluminum Boron Composites	104
IX	The Flexure Properties of 35 v/o B-Mg Composites with Various Span-to-Depth Ratios as Compared with Tensile Properties	106

#### SECTION I

#### INTRODUCTION

The principles and experimental aspects of fiber reinforced metals were admirably summarized in 1965 by Kelly and Davies<sup>(1)</sup> and Cratchley<sup>(2)</sup>. The first of those articles provided the basis for thinking about this new class of materials and the second served as a basis for believing in them. Together they constitute the point of departure for subsequent work both in England and in the United States. A large expansion in metal matrix composites research and development has occurred in the past four years. Cratchley<sup>(2)</sup> indicated that a "great deal remained to be accomplished in demonstrating the feasibility of fiber reinforced metals for various applications". He identified the design of components from metal matrix composites as the "one field of fiber reinforcement which had apparently received little or no attention". This critical review will address itself to the experimental progress which has been made toward the ultimate applicational acceptance of this new class of materials.

The historical development of filament reinforced metal matrix composites can be considered to initiate with the Jech, McDanels and Weeton(3), Sagamore Conference Paper in 1959. The reviews of Macklin(4) and Baskey(5) and the ASM Seminar volume(6) together with the Kelly and Davies(1) and Cratchley(2) papers provide a summary of the developments which occurred prior to 1965. Since that time two books have been published which are solely devoted to metal matrix composites (7,8) and multiple chapters of composite materials books are devoted to filament reinforcements of metals (9-18). A technical journal, Composite Materials, has been created in response to the expanding volume of research being generated. The defense Metals Information Center prepares a periodic Review of Recent Developments for the topic Fiber-Reinforced Metals and has issued two DMIC Reports (19,20) which summarize the research on many unclassified Government-sponsored fiber reinforced-metal research programs. The preceding references, the Survey of Ceramics Fibers and Fibrous Composite Materials (21), an ASM bibliography (22), a Defense Documentation Center report bibliography (23) and the NASA SCAN report notification service for the topic, Composite Materials, were utilized to identify over three hundred contributions to the technical literature pertaining to filament reinforced metal matrix composites.

The body of the review has been organized into three principal subdivisions in an effort to cope with breadth of content which the accumulated literature provides. The fabrication techniques section is intended to review the types of processes which have evolved for composite preparation, identify the forms of composite which can be generated by each process, define the level of mechanical properties which are representative of the application of specific processes to the various filament-matrix composite systems and identify the current limitations of each process.

The discussion of the mechanical properties of metal matrix composites is contained in the second section of the review. It deals with the character of those properties rather than the absolute values generated by individual experimentalists. And finally the problem

areas associated with or identified by the fabricational processes or mechanical properties are segregated for discussion. It is the objective of this final section to provide focus for future work by establishing priorities among the problems and suggesting experimental routes to their solution on the basis of the accumulated observations of the reviewed technical literature.

A historical observation of Thomas, Huffadine and Moore (24) regarding the development of cermets is most pertinent to the kind of development which must be accomplished for the composites field.

"In this particular field (cermets) the initial results rapidly gave rise to the realization that the problem of producing useful cermet combinations was more complex than had been originally envisaged. In particular, their brittleness and lack of impact strength became apparent. This factor, combined with the high cost and non-uniformity of much of the early material resulted in a waning of interest. The initial over optimism was replaced by undue pessimism. Throughout this phase the major emphasis had been placed upon the fabrication and testing of different metal/ceramic combinations in hope of finding a cermet with the desired properties. The amount of fundamental work done was, by comparison, small, and the net result was the accumulation of a mass of largely uncorrelated and inadequately understood data on very many different materials. Cermets were a new class of substrates with distinct characteristics and neither the basic mechanism of bonding, the effect of different modes of fabrication, the methods of testing required nor the special design considerations involved were sufficiently appreciated."

The problems of producing useful metal matrix composites has been identified to be complex and their high cost and nonuniformity are a caution flag relative to potentially waning interest. The initial optimism with regard to the achievement of a giant step forward in weight normalized strength and modulus has been fulfilled. The possibility of the development of undue pessimism can be avoided by the clear definition of the pertinent problem areas and the construction of an organized foundation of basic understanding beneath the technological advancement which has characterized these past four years of innovation. The composites field has its accumulation of uncorrelated and inadequately understood data but an appreciation has evolved for this new class of material which acknowledges the need for focused work on the basic mechanisms of bonding, the effect of various fabrication modes, the methods of testing and the special design considerations which are required.

#### SECTION II

#### SUMMARY

A variety of metallurgical processes including hot-pressure bonding, liquid-metal infiltrating, electrodeposition, vapor deposition, plasma spraying, cold press and sinter, extrusion and high energy-rate forming have been employed for the fabrication of filament reinforced metal matrix composites. Of these, by far the greatest emphasis has been placed on hot-pressure bonding techniques. The objective of any composite fabrication technique is to accomplish a specific form of material incorporating the reinforcing filament without breakage, with minimal reaction degradation, at a desired volume percent filament loading and with an interfacial bond which is sufficient to transmit the applied load from the matrix to the filament. Unfortunately the fabrication process development aspects of composite materials technology have not been reported in detail. Very few examples of processing parameter versus mechanical property data can be cited. The generalizations which have been reported are obviously based on such detailed studies but the process development effort has apparently been almost universally considered to be proprietary information.

The process development effort has consistently involved progress along a path of stated objectives:

- 1. Determine filament-matrix stability.
- 2. Achieve consolidation of filaments in a matrix.
- 3. Achieve tensile strength approximating that predicted by the rule of mixtures.
- 4. Reduce product variability at a high fraction of the rule of mixtures strength.
- 5. Achieve product scale up in size or flexibility of form.
- 6. Reduce processing costs.

Stage 3 objectives have been achieved for a multitude of filament-matrix combinations fabricated by a variety of processes and Stage 4 progress has been made for the most advanced composite system, hot-pressed aluminum-boron. The current applications oriented character of composites development centers on concurrent progress toward Stages 4 and 5 objectives. Processing cost reduction is an objective of the future which can only be achieved on the basis of a definable demand for a reliable material in a useful size and form.

Filament matrix stability is important to the development of a fabrication process for composite materials because it defines the degrees of freedom which exist for consolidation

with minimal degradation in filament strength properties. The only effective techniques for demonstrating filament stability is the pre- and post-fabrication mechanical testing of incorporated filaments. Optical microscopy, electron microscopy, microprobe analysis, electron diffraction, X-ray diffraction and microhardness scans have been used to identify the onset of gross reaction in a wide variety of filament matrix systems. Sophisticated analytical tools have been of little value in identifying the onset of degradation in filament properties.

The development of a sufficient interfacial bond to accomplish load transfer from the matrix to the filament is an intuitively obvious requirement to achieve rule-of-mixtures performance in the composite system. However, little has been done to define the magnitude of interfacial tensile or shear strengths, to determine what an adequate bond is or to correlate bond strength with composite mechanical property performance. The fractographic observation of adherent metal skins on filament pullouts, the coordination of pullout lengths with expected critical transfer lengths and the degree of filament fragmentation in the composite mechanical testing operation are qualitative indicators that a good bond is beneficial. However, extended time diffusion reaction which can develop a good bond can also result in filament strength degradation. The composite property optimization process involves the definition of the time-temperature regime which yields stability as measured by filament strength retention and the accomplishment of a well-bonded composite within that stability range.

Table I is a summary of the most prominently investigated filament reinforced composite systems relative to their fabricability in viable form by the various processing techniques. Figure I summarizes the representative forms of composite which can be fabricated by application of these fabrication techniques. Simple sheets or plates can be formed by hot-pressure bonding and utilized as honeycomb facing or creep formed to cylindrical or complex blade shapes. Casting processes can yield rod, tube or structural shape forms of composites which efficiently utilize the uniaxial character of composite properties. Deposition processes can provide circumferential reinforcement in the vein of cablewound cannon barrels. These processes can supply monolayer filament tape for wound vessel fabrication or to form complex shapes by laminate layup and pressing techniques.

The mechanical property potential of filament reinforced composites has been experimentally demonstrated for the most advanced composite system, Al-B. Rule of mixtures strength and modulus performance are illustrated in Figure 2a. The retention of tensile strength at elevated temperature is illustrated in Figure 2b as compared to the matrix material. The promise of dramatic improvements over available homogeneous aerospace materials in the applicationally important engineering properties of fatigue and stress rupture is demonstrated in Figures 2c and 2d, respectively.

These property summaries emphasize the maximum properties which have been attained with uniaxial materials but the ability to tailor the anisotropic behavior to provide the required level of desired properties for specific applications has been demonstrated in the published literature. These outstanding basic properties of filament reinforced metal matrix composites are accompanied by relative notch insensitivity and excellent vibration damping capacity. While it is easy to demonstrate the excellent properties of filament

Table I Fabricationally Stable Filament Reinforced Composite Systems

Fabrication Technique	Composite Systems	References
Liquid Metal Infiltration	Mg-B Cu-W Cu-Mo Cu-Ta Al-B Ni-W Ag-Steel Ag-W	25,26,58 27,28,87,104 28 29 34,79,111 56 125 125
Hot Pressure Bonding	Al-B  Al-Stainless Steel Al-SiC Al-Coated B Al-SiO <sub>2</sub> Mg-B Ti-SiC Ti-Be Ti-B Ti-Coated B	30,31,32,33,34,53,54,55,79,80,81,97,99,101,103 30,34,35,36,83,102 37,92,91,89,108,109 38 39 82,84,89,108,109,110 40 30,41 34 30,42 41
Cold Press and Sinter	Ni-W Ni-Mo Ti-Mo Ag-W	69 69 69 70
Plasma Spray	Al-B Al-SiC Al-Coated B W-W	32 39 39 66
High Energy Rate Forming	Al-B Ni-B Ti-B Al-W Ni-W Ti-SiC Ni-SiC	73,74 73,74 73,74 72,74 75.76

Table I (cont.) Fabricationally Stable Filament Reinforced Composite Systems

Fabrication Technique	Composite Systems	References
Electrodeposition	Ni-W	59,25,60,62,63,86
•	Ni-B	25,59,60.62,61
	Ni-SiC	38,26,62
	AI-B	65,62,27
	Al-SiC	38,26,62
	Cu-W	85,86,92
Chemical Vapor Deposition	· W-W	68
, .	W-B	68
	Al-Be	67
Extrusion and Rolling	Ni-W	69,88
	Ni-Mo	69
	Al-B	<i>7</i> 1
	Ti-Mo	69
	Ti-B	42

## Hot-Pressure Bonding

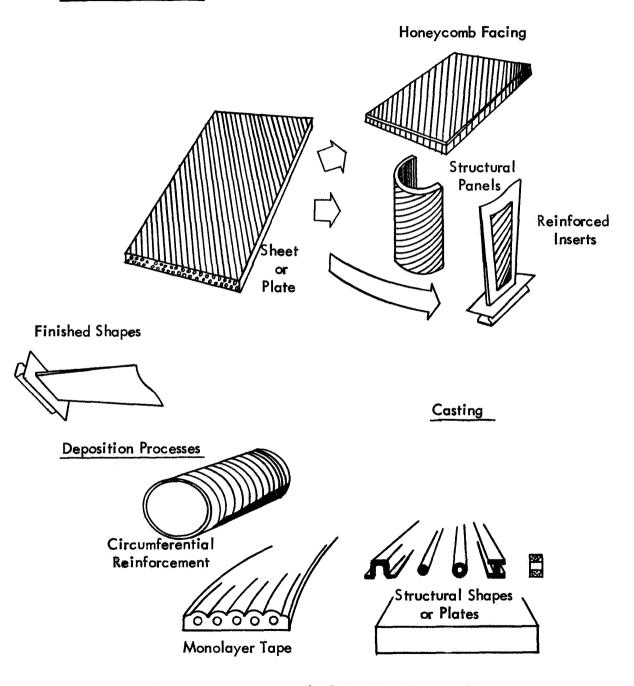


Figure 1. Composite Forms Which Can be Fabricated by Currently Available Processes

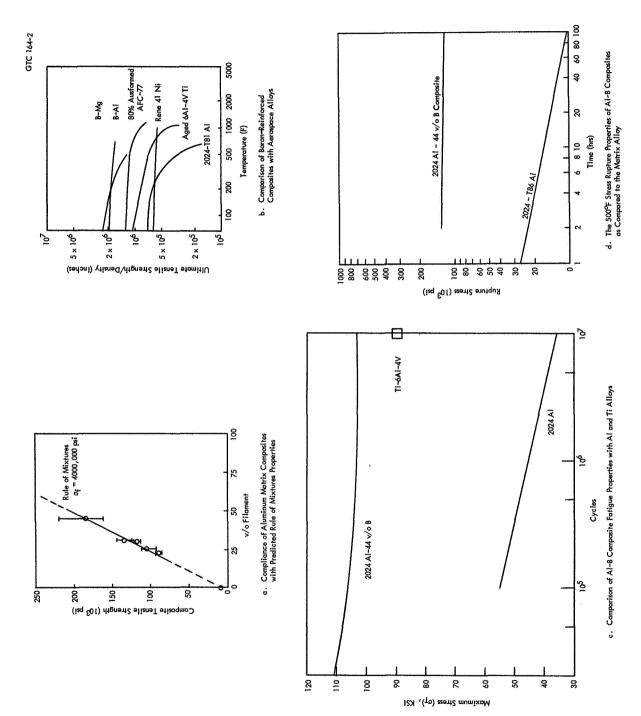


Figure 2. Demonstrated Properties of Advanced Metal Matrix Composites

reinforced metal matrix composites on the basis of selected values on specific systems, the accomplishment of the specific mix of properties required for a specific application represents the next step in the advancement of this laboratory material to the status of a structural element. That step involves both the detailed scientific study of the origins of those properties and the voluminous accumulation of the engineering data required to quality any material for high performance structural use. It requires the cooperative involvement of both the composite material engineer and the advanced structural design engineer.

The problems of filament reinforced metal-matrix composites fall into two basic categories:

- Those inhibiting design utilization of the demonstrated material properties
- 2. Those inhibiting the understanding of mechanical behavior

The former can be summarized as being consistency, cost and form (size and shape) while the latter encompass the construction of a scientific base of knowledge concerning the phenomenon which operate to yield the performance which has been empirically defined on state-of-the-art materials. In this latter area the model system studies utilizing Cu-W, Ni-W or Al-SiO<sub>2</sub> have begun to provide the framework for the development of understanding. The prerequisite to scientific studies of composite performance is a consistent starting material. Thus poor consistency which inhibits application is also a deterrent to meaningful examination of phenomenon involved in the performance of a composite material.

#### SECTION III

#### FABRICATION PROCESS DEVELOPMENT

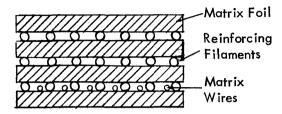
The need for ever increasing composite tensile strength data has taken precedence over detailed development of basic understanding relative to the various composite fabrication processes for at least three of the last four years. As a result high-strength filament reinforced samples have been fabricated to demonstrate the achievement of the predicted potential for metal matrix composite materials. The recent concentration on applications oriented materials development programs have underlined the need for improved reliability, lower cost and greater flexibility in product size and form as the three imperatives for future use. A comprehensive report on manufacturing methods for composite materials by Glasser and Sump(43) contained the metal matrix composite fabrication process development which preceded 1965 and summary articles by Sutton(44), Harmon(45), Davis(46), Thornton(47), Herzog(48), Alexander, et al.(49-51), Snide, et al.(52) and Weeton(93) record the continued progress in a general fashion.

#### 1. HOT-PRESSURE BONDING

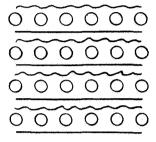
A variety of metallurgical processes, listed in Table I with applicable references, have been employed for the fabrication of composite materials. Of these, by far the greatest emphasis has been placed on hot-pressure bonding techniques. Included in the hot-pressure bonding category is any static pressure consolidation process carried out at elevated temperature whether it utilizes foil filament stacked arrays as in Figure 3a, sheet preforms as in Figure 3b, wire preforms as in Figure 3c or powder infiltrated spaced filament arrays as in Figure 3d.

The hot-pressure bonding process has been used successfully to fabricate aluminum, magnesium, titanium and nickel matrix composites. Foil filament arrays(11,18,20,21,33) are formed by carefully winding a specific filament spacing onto a foil covered drum and utilizing a cleanly decomposing binder to fix the filaments in place. An alternate technique(20) which accomplishes excellent control over filament spacing is cowinding of a matrix wire between reinforcing filaments. The foil filament array is removed from the drum and cut to the desired mat size for insertion into the hot press dies. A light retaining pressure is applied to the stack and the assembly is brought to pressing temperature with the attendent expulsion of the binder. The hot-pressure bonding step has been conducted in air, inert gas, or vacuum environments in chambers or as provided in a sealed retort.

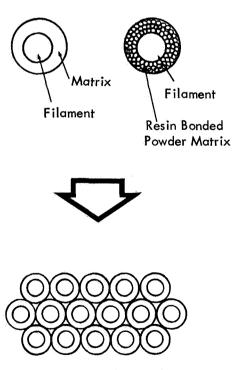
Tape preforms (23, 25, 38, 42) are handled in a similar fashion to the foil-filament arrays, Figure 3b. Both types of starting materials provide for the easy accomplishment of accurately oriented crossply filament orientations. The exhaustion of a binder phase is not a processing requirement for the matrix bonded preforms, however, the formation of the preform tape is considerably more costly than the preparation of a foil-filament arrays. Matrix coated filament, Figure 3c, has been successfully utilized (34,35,84) to form uni-axially oriented filament-matrix arrays. A systematic procedure for evaluating fabrication



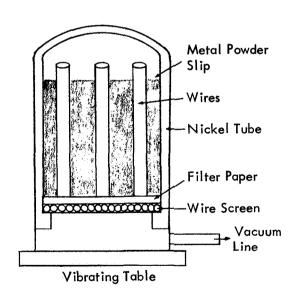
a. Metal Foil-Filament Array



b. Stacked Metal Matrix Tape Preformed



c. Matrix Coated Filament Array



d. Powder Slip Casting Apparatus

Figure 3. Basic Variations on the Hot-Pressure Bonding Process

parameters on the basis of longitudinal and transverse tensile tests together with shear tests and metallographic examination has been presented by Jackson<sup>(84)</sup> for the consolidation of Al melt-coated SiO<sub>2</sub> fibers. The larger degree of relative motion between filament in the consolidation step and the greater potential for random filament overlaps has resulted in the principal use of this process with the more ductile metal filament reinforced metal matrices. In such systems a considerable deformation of the incorporated filaments can occur especially as attempts are made for very high filament volume percent loading<sup>(35)</sup>.

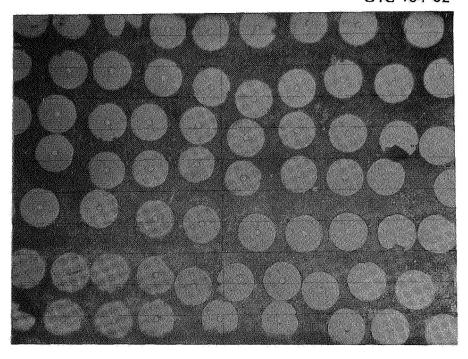
The final process of infiltrating spaced filament arrays with a powder metal matrix is the most tedious preparation procedure for the hot pressing of metal matrix composites, Figure 3d. The accomplishment and maintenance of good filament spacing is the tedious step. The resultant uniaxially aligned product can be consolidated by die or hot isostatic pressing and has characteristically been most successful with metal filament reinforcements. A similar preparation procedure has been utilized for use in extrusion consolidation or exposure to high energy rate forming processes.

The assembled preforms are exposed to a combination of temperature and pressure for a specific time in a controlled environment to yield the fully consolidated composite product. The hot-pressure bonding process has been most extensively utilized to form sheet or plate composite forms. Representative cross sections of hot pressed uniaxial and orthogonal crossply composites are presented in Figures 4a and 4b. The adaptation of the hot-pressure bonding technique to more complex shapes has been successfully demonstrated (53). The desired hot pressed final form is differentially simulated as shown in Figure 5a and pressed in resistance heated conformal dies, Figure 5b, to yield the desired complex shape, Figure 5c.

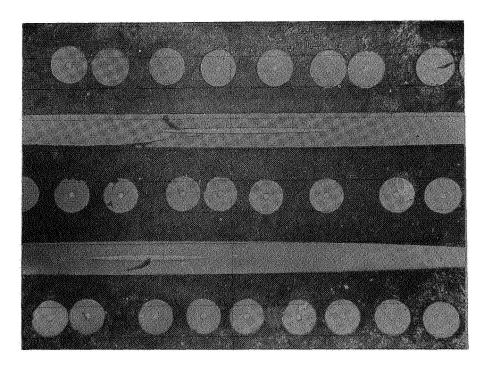
Optimization of the hot-pressure bonding fabrication process has been carried to the most advanced state for the aluminum-boron system as reflected by compliance of the maximum tensile strength properties as a function of volume percent filament with the strength calculated to be attainable from the proportional contribution of the incorporated filament and matrix (32,51,54,55). Filament strength degradation during an optimized hot-pressure bonding cycle approximates 10%.

#### 2. LIQUID METAL INFILTRATION

Some of the very first reinforced composites were produced by vacuum infiltration of a tube filled with filaments. Such techniques were and are applicable when the filament is liquid metal stable. The model systems of refractory metals in matrices which exhibit little mutual solubility represent a class of materials which have contributed significantly to the understanding of composite characteristics(27-29,56). SiO2 fibers have been successfully melt-coated by rapid passage of a single filament through a bead of molten aluminum by Jackson(84) and Wolff(34) has applied the technique to the formation of coated boron as a precursor for subsequent hot-pressure bonding. However, the extremely reactive nature of most advanced filaments has minimized the practicality of liquid state composite fabrication. The model systems have yielded reproducible quality composites at high volume percent filament loading with close correspondence to rule of mixtures strength and modulus predictions. Stability is the key to success in the utilization

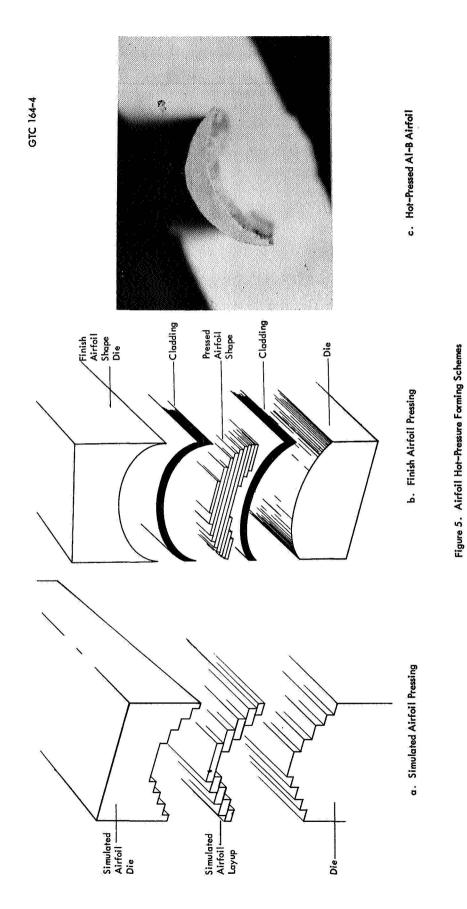


a. Uniaxial



b Orthogonal Crossply

Figure 4. Hot-Pressed Composite Panels



of liquid metal fabrication techniques and only magnesium of the structural metals shows significant stability in contact with boron to be practically fabricated by such techniques. Schuerch<sup>(26)</sup> achieved compressive strengths approaching 350,000 psi in early liquid magnesium infiltrated boron specimens. Wolff's work showed that only a few minutes of exposure to liquid aluminum is enough to seriously degrade the filament<sup>(34)</sup>. The documented stability of SiC in aluminum matrices<sup>(38,57)</sup> and the utility of coated boron (SiC, BN, or Ag) as a more stable reinforcement offers future potential for the expansion of liquid fabrication techniques. It is the relative instability of the available advanced filaments which has dictated that little effort be devoted to sophistication of liquid metal fabrication techniques.

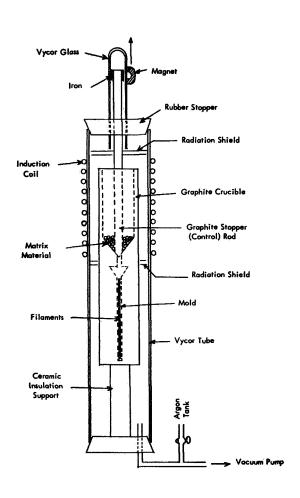
Vacuum infiltration, Figure 6a, of a bundle fiber in a tube is a batch process which is limited in size potential, and susceptible to incomplete or irregular fill. However, the utility of liquid infiltration techniques has been demonstrated by the continuous casting of boron filaments in a magnesium matrix (58) and by the effort to form three filament tapes by the rapid passage of boron through aluminum (34). The process simply involves the passing of a bundle of filaments through a metal bath in such a fashion as to accomplish wetting of the individual fibers as they enter the bath and wiping off the excess as the bundle is drawn through an orifice in the bottom of the crucible, Figure 6b. The simplicity of the process suggests that volume production of rod, tube or structural shapes is possible at little added cost over that of the incorporated raw materials. The microstructure of a 75 v/o boron-magnesium continuously cast rod is shown in Figure 7a while Figure 7b shows the almost perfect hexagonal packing of the most densely packed areas.

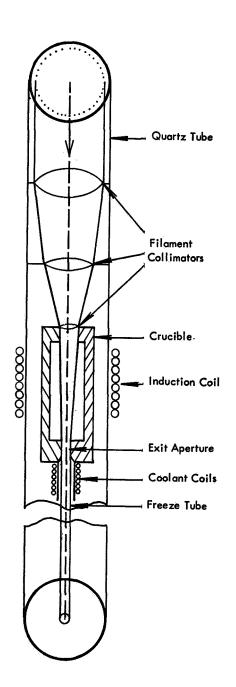
The continuous casting process is capable of yielding any uniform cross section form of uniaxially aligned filament reinforced composite. Rods, tubes and structural shapes are uniaxial forms of material and as such can take full advantage of the maximized composite properties in the axial direction. Unidirectional strength-to-density values in excess of  $2 \times 10^6$  inches and modulus-to-density ratios in excess of  $500 \times 10^6$  inches place this type of material in the position of exhibiting a two to four times advantage over conventional aluminum and titanium alloys.

#### 3. ELECTRODEPOSITION

The utilization of electrodeposition as a fabrication technique saw early application with reactive filaments because it could be accomplished without elevated-temperature exposure and a concurrently deposited sample of matrix could be obtained for mechanical test (59-63). The electroforming technique involves the electrodeposition of the matrix onto a suitable mandrel while concurrently winding the filament reinforcement and has been discussed by Bonnano (64) and more recently by Baker (63). A schematic of the composite fabrication process is shown in Figure 8. The technique is applicable to any metal that can be electrodeposited and has the following advantages:

- 1. It is a room temperature fabrication process.
- 2. A fully dense matrix sample can be concurrently deposited.
- 3. Intimate filament-matrix contact is accomplished at the interface.





6a. Vacuum Infiltration

6b. Schematic Representation of Continuous Casting Process

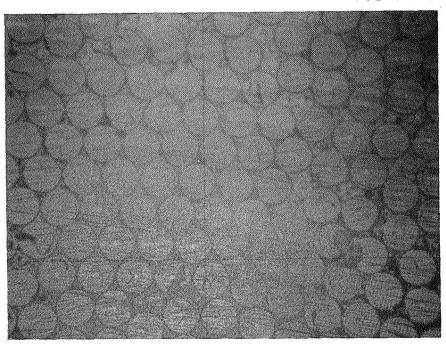


Figure 7a. Typical Cross Section of a Continuously Cast Rod

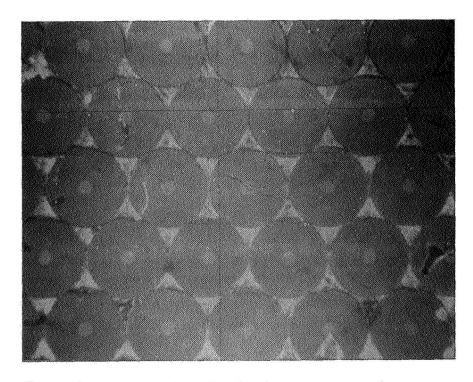


Figure 7b. Cross Section of a Continuously Cast Rod Showing Almost Perfect Hexagonal Packing of Filaments

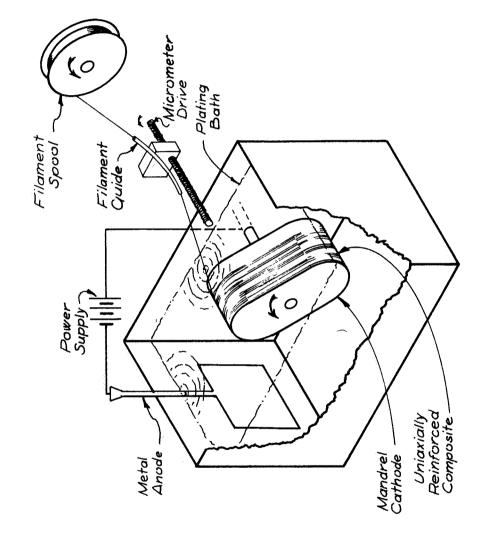


Figure 8. Schematic of the Electrodeposition Process for Forming Aligned Filament Composites

- 4. Any shape which can be made as a surface of revolution can be fabricated.
- 5. Accurate control can be exercised over filament spacing and thus volume percent loading.

Figure 9 is a schematic of the growth pattern which is characteristic of this fabrication process. Figure 9a indicates the mode of formation of the electrodeposit on the filaments which are wound onto an undercoat of nickel on the winding mandrel. Figure 10 shows a monolayer nickel-boron tape formed in this manner. Figure 9b shows the continuation of the process by winding and coating of a second layer. Multiple layer samples are produced by a repetition of this process until the desired thickness is achieved, Figure 11. Figure 9c shows the location of potential void sites in the composite structure. Type 1 voids occur when deposition on the filament progresses at a rate such that the growth from two adjacent filaments intersects before growth from the undercoat reaches the point of intersection. Type 1 voids can be grown out at wide filament spacings by flooding the mandrel with fresh electrolyte and by the imposition of plate-deplate cycles on the forming operation. Type 2 voids are formed when the surface contour of the overcoat for the first layer does not conform to the filament size and shape. The character of such voids is shown in Figure 12. If the overcoat is thick enough the grooves become rounded and accept the subsequent filament layer with little porosity. However, at high volume percent loadings the crevices occupied by the circular filaments leave triangular voids beneath them. It should be emphasized that even when the geometry of the surface is correct for the acceptance of the filament without void formation, the character of the bond between the filament and the matrix is different at the contact point than on the rest of its circumference. At that point it has simply been laid against the matrix, while elsewhere the matrix has been electrodeposited onto it. Another important consideration in the characterization of electroformed composites is the need for accurate control over filament spacing. Variations in filament spacinas result in changes in the surface contour of the electrodeposited overcoat. Wide spacings yield a larger valley and close spacings create a larger hump and the effect of such misspacings is to force greater misspacings upon the subsequent layers. Such misspacings ultimately lead to a greater void formation in higher volume percent multilayered specimens.

This effort characterizing the electroforming process for continuous filament reinforced composites can be summarized as follows:

- 1. Monolayer filament tapes can be produced with minimal void entrapment to roughly 45 v/o.
- Multilayer composites can be formed to equivalent volume percent loadings but geometrical considerations combined with the potential for misspacings make void formation a problem to be contended with.
- 3. A densification process should be considered necessary in conjunction with composites formed by electrodeposition.
- 4. Monolayer tapes can be used as a raw material for multilayer composite fabrication by hot-pressure bonding.



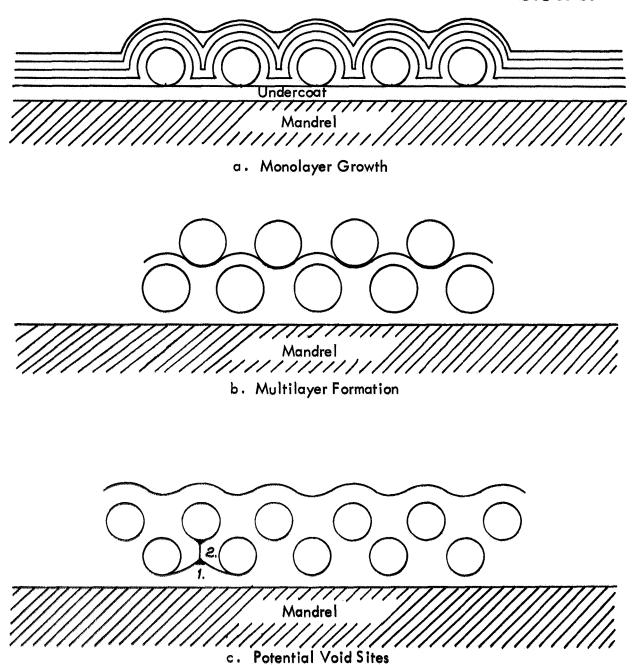


Figure 9. Schematic Representation of Electroformed Composite Formation

# GTC 71-6

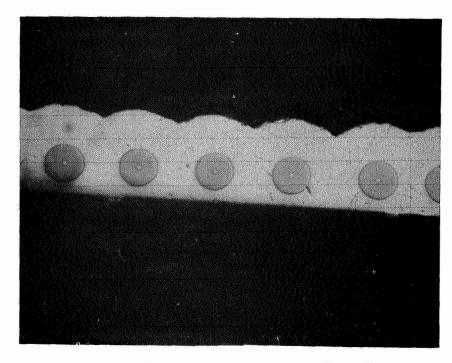


Figure 10. Filament-Reinforced Monolayer Tape

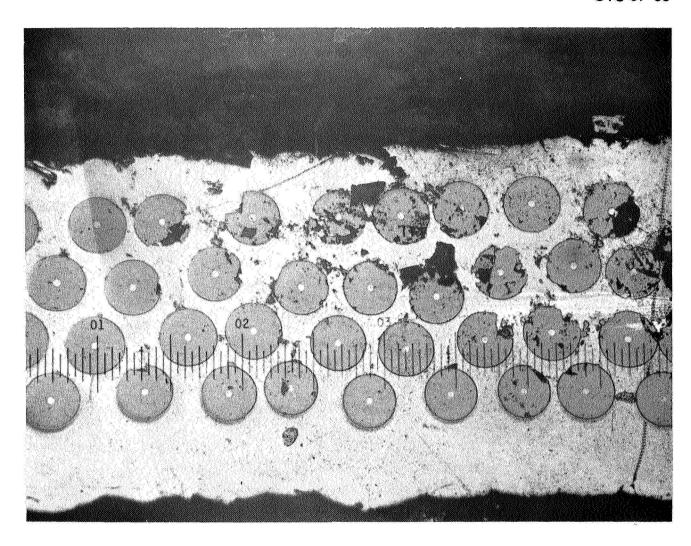


Figure 13. Cross Section of a Typical SiC-Ni Composite, Specimen #27, as Polished, (150X)

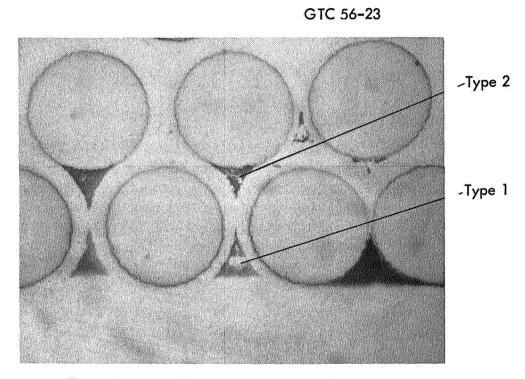


Figure 12. Void Formation in Ni-W Composites

In addition to monolayer tapes, multilayer circumferentially wound reinforced structures can be formed as illustrated in Figure 13, a 20 filament layer circumferentially sound simulated motor case in the Al-B system. The multilayer circumferentially wound composites have demonstrated a tensile insensitivity to the hoop oriented voids  $^{(65)}$  as shown in Table II. Hoop strengths exceeding 200,000 psi with a modulus of 35 million psi generate strength-to-density values in excess of  $2.0 \times 10^6$  inches and modulus-to-density values over  $400 \times 10^6$  inches. Composite strength in the transverse direction is minimal (20--30%) of the matrix strength because of the incorporation of 10--15% voids.

The electrodeposition process is capable of yielding a continuous monolayer composite form of a width which is only limited by the engineering ability to collimate thousands of filaments. Circumferentially wound structures of several feet in diameter could be deposited as a simple extension of demonstrated fabrication capabilities.

#### 4. PLASMA SPRAYING

The plasma spraying technique for composite fabrication has been optimized most completely by Kreider<sup>(32,39)</sup> in the aluminum matrix system and the feasibility of the process for the formation of tungsten wire reinforced tungsten rocket nozzle configurations has been demonstrated<sup>(66)</sup>.

The process is shown schematically in Figure  $14^{\left(32\right)}$ . This schematic is equally applicable to the description of chemical vapor deposited and vacuum deposited matrices on which a lesser degree of development has been conducted. A layer of matrix in the form of a foil or as a plasma sprayed layer on the mandrel is overwrapped with a spaced array of reinforcing filaments which are incorporated by the spraying of a subsequent layer of matrix. The operation is conducted in an inert atmosphere or with a protectively-shrouded flame. The as-sprayed matrix is not fully dense (12-15 v/o voids) and contains a somewhat higher oxide content (1.5 wt %) than foil type material. Transverse strength is low in the as-sprayed condition and matrix ductility is lower than wrought aluminum. The impact of molten particles of matrix on the filament surface provides intimate contact on the filament matrix interface, and the immediate quenching of the small particles prohibits extensive reaction degradation in the composite formation process. Boron degradation is approximately  $20\%^{\left(32\right)}$  in the plasma spraying process while the coefficient of variation is almost doubled.

The preparation of multilayer composites by plasma spraying is characterized by the same sort of void formation and filament misplacement difficulties described for the electrodeposited composites. Also, circumferentially reinforced composites exhibit the same poor transverse properties. Post deposition sintering treatments spherodize voids but do not increase matrix density while hot-pressure bonding of multilayer or stacked monolayer plasma sprayed material results in a fully consolidated composite with transverse properties approximately 1/2 that of the matrix itself.

Further optimization of the plasma spray composite fabrication process<sup>(39)</sup> has been achieved utilizing the silicon carbide coated boron filament which exhibits significantly less sensitivity to the high oxygen content of the sprayed matrix. Coated boron filament

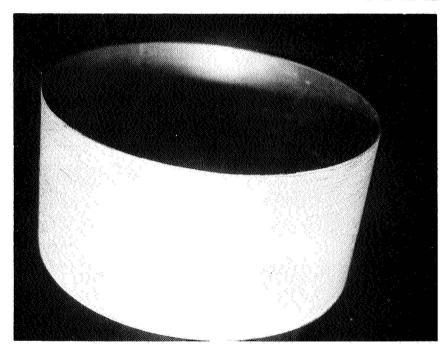


Figure 13. Motor Case Section of Filament-Wound Boron in Aluminum Matrix

Table II Tensile Properties of Electrolytically Deposited AI-B NOL Rings

Run	Number of Layers	Composite Density (g/cc)	Filament v/o	Void v/o Calculated	Composite Strength (10 <sup>3</sup> psi)	Composite Modulus (10 <sup>6</sup> psi)	Composite Strength/ Density (10 <sup>6</sup> in.)	Composite Modulus/ Density (106 in.)
NOL-23	<b>,</b>	2.22	25	16.5	56	1	0,70	8
NOL-24	.23	2.22	29	16.5	8	4	1.01	{
NOL-34	က	2.29	38	14	83.1	20°9	1 .00	252
NOL-29	ଳ	2.27	4	16	87.4	24.4	1 .07	298
NOL-31	2	2.20	53	17.5	127	28.7	1 °60	361
NOL-28	2	2,32	55	14	113	26.9	1 ,35	321
NOL-32	4	2.48	28	7	137	36.2	1.53	404
NOL-35	ო	2 . 40	58	10	135	34.1	1.56	394
NOL-33	10	2.26	99	15	164	31.8	2.01	390
NOL-37	10	2.26	70	15	189	34.5	2.31	422

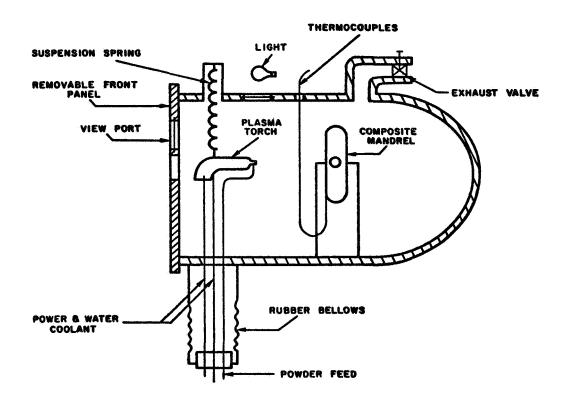


Figure 14. Plasma Spray Process

does not exhibit the relatively large fabrication degradation nor does it degrade as rapidly as a function of time at temperature in the plasma sprayed condition. This effort concentrated on the optimization of the plasma spraying process to yield a tape preform for subsequent hot-pressure consolidation.

The monolayer tape material as a preform for subsequent hot-pressure bonding experiments has an operational advantage over organically bonded foil filament arrays in that no binder need be exhausted in the fabrication step. Commercially available steel foil heat treating envelopes can be utilized to provide the protective environment for the hot-pressure bonding step. However, the higher cost of the preform fabrication process, the necessity of using a higher price coated filament, and the effect of the higher matrix oxide content on stability and mechanical properties are identified disadvantages.

The excellent as-sprayed mechanical properties qualify this process for the formation of multilayer circumferentially reinforced bodies such as the simulated motor case shown in Figure 11 or as a hoop reinforcement in the intermediate temperature ring sections of jet aircraft engine compressor analogous to the boron reinforced resin rings which have been evaluated by Pratt & Whitney at lower temperatures.

A direct application evaluation for plasma sprayed composites was conducted by Greening (66) where tungsten fiber was incorporated in a tungsten matrix by plasma spray deposition. Figure 15 shows a mandrel with a helically wrapped array of filaments prior to plasma spraying. Operational tests on the fabricated nozzles indicated the feasibility of this high-temperature refractory composite structure for relatively simple fabrication of many complex configurations on conventional equipment at a nominal cost.

# 5. CHEMICAL VAPOR DEPOSITION

Chemical vapor deposition as a technique for analogously infiltrating filament arrays have been investigated in a preliminary fashion. Withers (67) has worked with the Al-Be system utilizing the decomposition of aluminum alkyls on a heated mandrel wound with beryllium filament. While chemical vapor deposition yields a fully dense deposit of the matrix, the irregular nature of the composite surface after the encapsulation of the first filament layer causes misspacings and contact voids as subsequent layers are added. Chemical vapor deposition has the process attribute of yielding metal deposition on all heated surfaces simultaneously rather than being a line of sight process as in plasma spraying or depositing only on the surface of electrically conducting constituents as in electrodeposition. The chemical vapor deposition process operates at a temperature which is low relative to the metal melting point or the effective hot-pressure bonding temperature and thus offers the potential of nonreactive consolidation. While feasibility has been demonstrated, fabrication process optimization would be required to achieve useful mechanical properties in a filament wound structural application.

The feasibility of forming W-W and W-B composites by chemical vapor deposition techniques has been demonstrated by Greszczuk<sup>(68)</sup> where the relatively low metal deposition temperature (800-1100°F) made consolidation possible without the severe reactivity that would have accompanied hot-pressure bonding or liquid infiltration procedures. The

# GTC 164-64

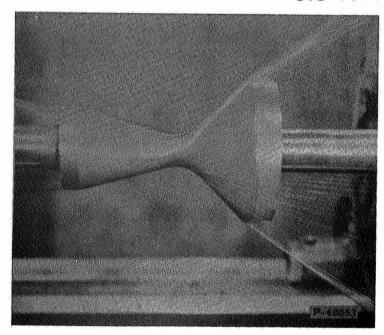


Figure 15. Application of Helical Wrapping of Tungsten Wire to Mandrel

deposition morphology reported indicates that continuous tape formation by chemical vapor deposition on a spaced array of filaments can be considered feasible.

## 6. COLD PRESS AND SINTERING

The process of cold pressing powder-filament arrays and sintering has been utilized principally as a preparation procedure for subsequent extrusion and rolling consolidation. The process has been principally applied to metal filament reinforcement of metal matrices. Baskey<sup>(69)</sup> has indicated that the long time sintering of cold pressed powder-filament blends at relatively high temperature is an undesirable technique for the accomplishment of full density composites because of excessive degradation of incorporated filaments. Additionally the thermal expansion mismatch in some systems, i.e. W or Mo in Hastelloy X results in the matrix expanding away from the wires during sintering. Thus cold pressing or cold pressing and sintering were used only to achieve compact and coherent billets for subsequent hot pressing, extrusion or rolling. Adamski, et al.<sup>(70)</sup> on the other hand utilized the cold press and sinter technique to form Ag-W composites with mechanical property results which were equivalent to hot pressed samples. However, the relatively small number of specimens involved make speculation as to the influence of fabrication variables on properties most hazardous.

The Baskey observation that long-term elevated temperature exposures are required for a high degree of densification would seem to limit the process to filament-matrix combinations where extreme stability is exhibited, i.e. stable reinforcements in the low melting metal matrices.

### 7. EXTRUSION AND ROLLING

Refractory metal wires have been consolidated in nickel base superalloys are in titanium alloys by extrusion and rolling techniques (69) and the brittle filament boron has been extruded in an aluminum matrix (71). The Baskey (69) work demonstrates how thorough a fabrication process development program can be when a relatively inexpensive filament is being incorporated by a production type process. The experimental program defines the chemical compatibility of the alloys and filaments of interest and then proceeds to fabricate discontinuous and continuous filament composites from hot or cold pressed billets by extrusion and hot rolling within the defined range of chemical compatibility. It is one of the few detailed documentations of the influence of fabrication variables on the properties of fiber-reinforced metals.

It is clearly demonstrated that randomly oriented discontinuous filament-matrix powder mixtures can be cold or hot pressed into a preform for extrusion to yield uniform cross section rod composites containing aligned fiber reinforcements. The effects of preform treatments and extrusion parameters together with post-extrusion heat treatment upon the broadest possible range of pertinent mechanical properties are examined with a sufficient number of samples to permit the definition of a reliable set of conclusions which define the optimum treatment for particular sets of desirable properties. The results are compared to both the unreinforced matrix similarly treated and to the wrought forms of alloys which are the conventional competitors for the applicational range envisioned for these composite materials. Initial filament strengths are monitored but the composite is characterized in terms of what processing variations can do to its properties rather than in terms of what theory predicts it should be capable of accomplishing. Improvements are registered in both the nickel base superalloys and in the titanium alloy matrices in tensile and yield strengths and stress rupture properties formed by extrusion of discontinuous refractory metal filaments reinforced powder compacts. Continuous filament composites of equivalent volume percent loadings exhibited better performance than the discontinuous ones. Very well bonded composites were achieved in spite of the large disparity in thermal expansion coefficients.

### 8. HIGH ENERGY RATE FORMING

The utilization of high energy rate forming as a composite fabrication technique has been surveyed by a number of investigators at the Pacific Northwest Laboratories of Battelle Memorial Institute. Figure 16 is a schematic of pneumatic impaction apparatus utilized to impart pressure pulses of up to 400,000 psi to canned powder filament arrays inserted in the die at elevated temperatures. While the process has admirably demonstrated its capability to accomplish consolidation there has been little process optimization against filament degradation or composite properties. The process feasibility work has indicated that low consolidation temperatures result in excessive filament breakage and it is apparent that higher consolidation temperatures result in serious degradation in filament properties. It is apparent from the mechanical property data on the system which has been studied in the most detail, Ti-SiC(75,76), that the time-temperature exposure associated with the high energy rate forming step is excessive. The optimum HERF temperature of 11:00°C for that system is significantly above the 900°C maximum temperature utilized for hot-pressure bonding consolidation of the same system (38,41).

The justification for detailed HERF process optimization is a useful metal matrix system lies in the requirement for scale up in the size of sheet or plate composites which can be reproducibly fabricated. In principal, the use of explosive techniques of HERF to apply the consolidation pressure for large size composites is an attractive one. The serious problems of maintaining a uniform distribution of filaments while minimizing both filament breakage and filament reaction degradation have been identified and serve as the basis for continued exploration of the utility of this composite fabrication technique.

The summarized processes represent the source of various forms of composites. The most advanced yield properties which closely approximate the values predicted by rule of mixtures calculation. The accomplishment of rule of mixtures values is heralded and broad generalizations concerning the effects of processing variables on the attainment of such properties are recited. But the experimental data which document the generalizations are largely invisible.

The accomplishment of high strength and modulus values has experimentally demonstrated the potential for composite materials but the translation of existing processes to useful size, with good reliability and at a cost competitive with more conventional alloy designs is the current challenge to the experimentalist. What then can be derived from the critical evaluation of the reported experimental accomplishment in the area of composite fabrication technology?

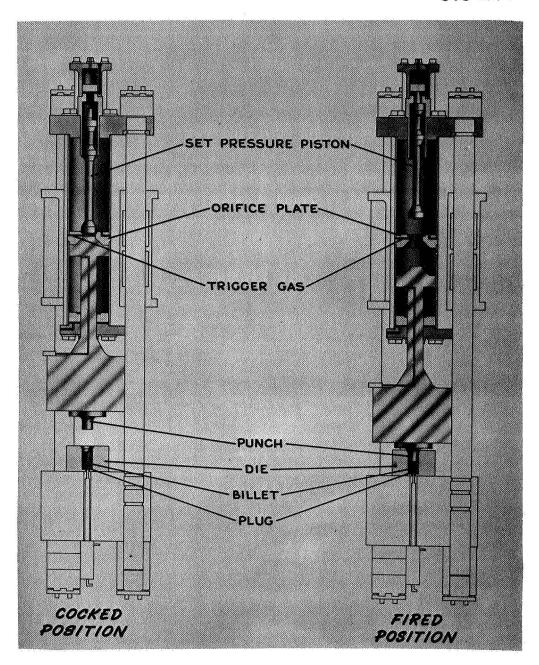


Figure 16. Schematic of Pneumatic Impaction Machine Used to Consolidate Fiber Reinforced Composites

Table III is a summary of the types of process development data which has been most informative in the dual task of optimizing composite mechanical properties and understanding the origins of the derived properties. Contained in Table III is the blueprint to the understanding of product variability. In simple terms we must understand the filament reinforcement that is being utilized; we must understand it in terms which are pertinent to its functioning in the composite; we must monitor the effect of various processing variables upon its characteristics; and we must correlate its characteristics with the properties derived from the fabricated composite. On a parallel framework we must understand the load internal translation process in a functioning composite in terms of the character and strength of interfacial bond which is developed by the range of fabrication parameters which maintain filament properties and accomplish composite consolidation. The model for composite failure outlined by Rosen (77) defines the type of data which is essential to the establishment of consistency in such materials:

"When a fiber break occurs, there are several possibilities for the subsequent behavior of the composite. First, the high interface shear stresses may produce interface failure which could propagate along the length of the fiber reducing the fiber effectiveness over a substantial fiber length. To achieve the potential of the fiber strength, it is necessary to study and determine the fabrication conditions which will yield an interface sufficiently strong to prevent interface shear failure. This can be done by using either a high-strength bond or a ductile matrix which permits redistribution of the shear stresses. In the latter case the lenath of fiber which is affected by the break will increase as it will take a longer distance to retransmit the stresses back into the fiber at the low stress level of a ductile matrix. With a strong bond, the interface conditions can be overcome as a potential source of failure. Second, the fracture toughness of the matrix must be considered to prevent the propagation of a crack through the matrix and parallel to the filaments. A third possibility is that the initial crack will propagate across the composite resulting in failure. This is influenced in part by the fracture toughness of the matrix, and again, since it is clear that with brittle fibers one can always expect a fracture to occur at a relatively low stress level, it is important that the fracture toughness of the matrix material is sufficient to prevent propagation of this crack across the composite. If these two potential modes of failure are arrested, it will then be possible to continue to increase the applied tensile load and to obtain breaks at other points of imperfection along the fibers."

Thus the ultimate objective of a metal matrix composite development program must be a sufficiently well-bonded system to accomplish repeated filament fracture until a statistical accumulation of fiber fractures occurs in the vicinity of one cross section to provide the opportunity for final catastrophic failure. While the rule of mixtures has been an admirable target for composite fabrication optimization, every major fabrication program has yielded individual composite strength values which exceed rule-of-mixtures calculations. If such strength values are to be attained consistently, the statistical nature of the failure phenomenon must be realized and new objectives must be set which transcend those which would be calculated from a simple rule-of-mixtures calculation.

Table III Types of Data Utilized to Evaluate Fabrication Process Development

Kreider(32)  Kreider(33)  Young(53)  Young(53)  Lenoe(79)  Sumner(37)  Sumner(37)  Sumner(37)  Shimizu(53)  Hoffmanner(35)  Hoffmanner(35)  Hoffmanner(35)  Kreider(39)  Hoffmanner(35)  Kreider(39)  Adamski(70)  Hoffmanner(35)  Miller(75)  Dean(56)	<pre></pre>	* * * * * * * * * * * * * * * * * * *	* * * * * * * * * * * * * * * * * * *
Parameter (30)		gth) with U.T.S.c	r (Processing t & 1) f (Processing t & p) f (Processing T & p) f (Processing T & p) Qualitative Evaluation of Bonding Quantitative Evaluation of Bonding Minimization of Filament Degradation Maximization of Composite Strength Minimization of Composite Variability

We know that filament strength goes up with decreasing gauge length<sup>(78)</sup>. We know that the standard deviation on strength goes down with decreasing gauge length<sup>(79)</sup>. We know that filaments do break to relatively short lengths in tested composite specimens<sup>(39)</sup> and we know that the filament fragment lengths tend to be shorter immediately adjacent to the site of final specimen failure<sup>(41)</sup>. The quantitative representation of these characteristics as a function of fabrication parameters is essential to the basic understanding of composite fracture and minimization of variability in composite specimens.

The work of Lenoe<sup>(79)</sup> establishes statistically the ultimate tensile strength of the filament utilized in that program as a function of gauge length. The standard deviation on U.T.S. as a function of gauge length could be computed. The thermal degradation of the filament for time-temperature exposures which were pertinent to the fabrication procedure was documented in sufficient detail to again permit calculation of standard deviations on U.T.S. at a single gauge length. Filament extracted from fabricated composite samples were tested in the same fashion. The expansion of this procedure to cover a range of gauge lengths and to document the effect of the matrix digestion solution on heat treated filament would provide the background for the interpretation of digested filament degradation effects as a function of fabrication time, temperature, pressure and environment as reported by Cunningham (33) or the correlation of filament degradation data with composite properties as a function of complete range of processing parameters such as were studied by Baskey<sup>(69)</sup>. The accomplishment of minimum filament degradation in the fabrication process and the utilization of the full strength contribution of the minimally dearaded filament requires an adequate bond at the filament-matrix interface. Thus quantification of the measurement of the degree of shear strength developed at the interface is essential. The length of sheathed filament pullouts or the measurement of post tensile test filament fragments in the specimen gauge length are techniques which have been utilized. The consistent minimization of filament strength degradation and the concurrent establishment of an adequate degree of bonding at the load transfer interface will permit the evaluation of composite fabrication processes on the basis of consistent full utilization of reinforcing potential.

Cost is the second deterrent to early application of metal matrix composites on a competitive basis with other structural materials. The \$500-\$600/lb price for hot-pressure bonded material in multi-thousand pound quantities represents fabrication costs which are a factor of 3 to 4 over the cost of incorporated filament. Process economization is obviously essential. Many metallurgical processes can and will prove capable of yielding viable composite samples but detailed and expensive process development effort must give heavy emphasis to the potential for economical volume production in a useful size.

#### SECTION IV

# THE MECHANICAL PROPERTIES OF FILAMENT REINFORCED METAL MATRIX COMPOSITES

### 1. TENSILE PROPERTIES AT ROOM TEMPERATURE

It is impossible to discuss the tensile properties of composite materials without introducing the "rule-of-mixtures". The rule-of-mixtures is many things to many people, but it is almost always the line through the experimental points generated in any particular program. The abuses of the rule-of-mixtures are so gross that it is really essential to define the rule-of-mixtures before discussing the tensile properties of metal matrix composites.

and it is assumed that the strains  $\epsilon_m = \epsilon_f = \epsilon_c$  for matrix, filament and composite are equal.

The rule-of-mixtures can be a useful first approximation of composite performance. It can also be utilized for rationalization.

The volume fraction filament,  $V_f$ , must be the actual volume percent of filament in the tested cross section of the sample. The tensile strength of the filament must be the actual strength of the incorporated filament at the gauge length that it breaks to before ultimate failure of the composite specimen. The stress in the matrix  $\sigma_m^*$  must be the stress which is sustained by the matrix at composite failure. It must take into consideration the modifying effect of possible alloying with the reinforcing filament or of residual or developed states of stress characteristic of its use in composite form.

A nominal V<sub>f</sub> cannot be used honestly as a measure of composite performance. Likewise one cannot be casual about the determination of V<sub>f</sub>. If 800 filaments are counted in a 1/2"  $\times$  .040 inch tensile specimen cross section and a 4.0 mil diameter assumed, the V<sub>f</sub> = .502 but if the filament is barely within specifications at 3.9 mils then the V<sub>f</sub> = .477, a difference of over 10,000 psi in calculated composite strength. The measurement of filament volume fraction by weight in the matrix digestion process utilized by Schaefer (97) or the metallographic technique described by Taylor (100) seem to be the most reliable measures of V<sub>f</sub>.

The tensile strength of the filament must be that strength which statistically represents the filament as incorporated into the composite. If the filament is degraded upon

fabrication then it is unfair to insert its virgin strength. Similarly if it breaks to 1/4-inch segments in the composite tensile test it is unfair to utilize the 6-inch gauge length filament strength value. Grinius (78) has indicated that boron filament is roughly 10% stronger at a gauge length of 0.3 inch than at 1.0 inch. Most rule-of-mixtures calculations are based on an average strength measured at a 1 inch aguae length while the critical load transfer length would be less than 0.1 inch for boron in the aluminum matrix. Thus the rule-of-mixtures calculation would be conservative by the difference in filament strength contribution calculated for the 1 inch and < 0.1 inch fiber. An observation by Rosen(77) points out that a very variable lot of filaments at 400 ksi average 1-inch gauge length strength would have a steeper slope to its gauge length vs strength plot (Figure 17) and thus would have a much greater average strenath at the 0.1 inch length than would be characteristic of a very consistent filament lot, i.e., almost constant plot of strength as a function of gauge length. Thus, contrary to the many superficial explanations of low composite strength, a high average strength filament with a high standard deviation should yield higher composite strengths than a similar average strength material with a minimal standard deviation, if Rosen's postulations are correct.

In spite of direct experimental evidence (hardness measurements) that reaction between filament and matrix is strengthening the matrix,  $\sigma_m^*$  values for the virgin metal are universally applied to the rule-of-mixtures calculation and almost as universally the benefit is attributed to the filament. Thermal expansion mismatch between matrix and reinforcing filament creates an undefined state of residual stress in the composite at room temperature for any specimen fabricated at elevated temperatures. The magnitude of those residual stresses depend upon the relaxation properties of the matrix as well as the thermal expansion coefficients. Herman<sup>(41)</sup> has presented a phenomenological treatment of the influence of a residence state of stress or composite tensile behavior. Simply stated the lesser expansion coefficient of the filaments (B or SiC) relative to the matrix (Al or Ti) can yield residual tensile stresses in the matrix, balanced by residual compressive stresses in the filaments. The effect of the tensile stress in the matrix can be seen in Figure 18 to shift the matrix stress strain curve 1 to the left, which results in an increase in the ultimate strength of the matrix at the failure strain of the filaments. It also shifts the composite proportional limit,  $\sigma_{cp}$ , (due to the onset of plastic flow in the matrix) to a lower strain. The residual compressive stress on the filament shifts the filament stress-strain curve 2 down to provide an additional increment of composite tensile strain, efr, before filament ultimate strength is reached to cause failure. This treatment assumes that the tensile strain on the filaments determines the composite failure strain, since it is impossible to attribute a strain increment to the elongation which takes place as a result of load transfer in the matrix after filament fracture begins to occur.

An X-ray diffraction technique for the in-situ measurement of the lattice strains in the filament and matrix constituents of a composite has been developed by Heckel (94,95). It is a valuable tool for the quantitative determination of the residual stresses in a composite and can be utilized to evaluate the load transfer mechanisms during the application of load to composite specimens. The matrix residual stresses in hot pressure bonded Al-17.5 v/o W were found to be relatively low while compressive residual stresses in the filaments ranged from 50,000 to 75,000 psi in the as-fabricated condition. Composite yielding corresponded to yielding in the matrix for all composites tested. The yield stress values



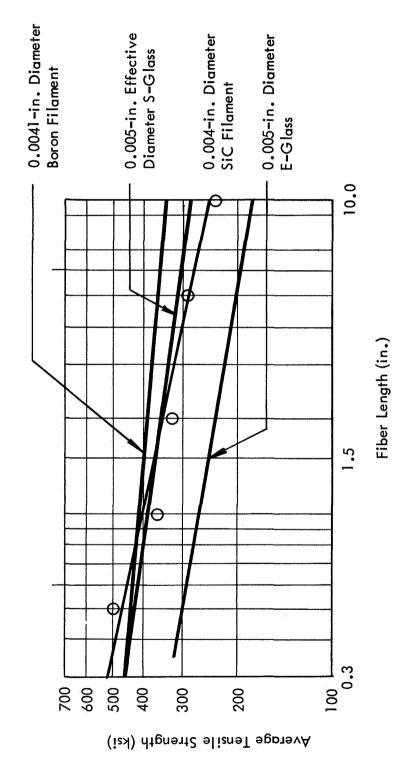


Figure 17. Effect of Specimen Length on the Tensile Strength of SiC Filaments

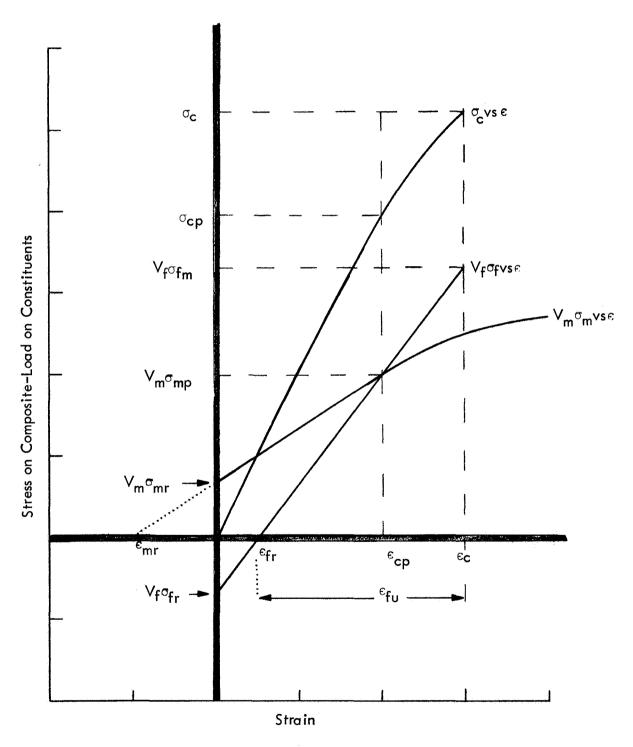


Figure 18. Composite and Constituent Stress-Strain Curves with Tensile Residual Stresses in the Matrix

calculated from the total lattice strain corresponded to the yield values reported for fully annealed 2024 aluminum regardless of the original residual stress level. Such an informative experimental technique should be coordinated with the metallographic, microhardness and electron scanning microscopy approach to composite deformation which has been reported by Jones (115).

There has been little mechanical property testing which can contribute to the clarification of the effect of residual stresses on composite performance. Schmitz<sup>(42)</sup> has experimentally identified a proportional limit shift in stress relieved titanium matrix composites which corresponded with the predictions of the model. The works of Sumner<sup>(98,99)</sup> treat residual stresses as implied by a variety of heat treatments on companion composite samples in the steel and boron reinforced aluminum systems respectively. In the steel reinforced system which exhibits a poor interfacial bond the stress-strain diagrams for both annealed and for heat treated and aged specimens fall below the theoretically calculated curves in accord with the predicted effect of relaxation of thermal expansion mismatch strains in the matrix. Boron reinforced composites evaluated in the same program yielded contradictory results. Stress relieved specimens exhibited improvements in both ultimate tensile strength and consistency. Unfortunately the effect of the various heat treatments on the alloy matrix as modified by any alloying with the filament is an unknown factor which clouds the unequivocal interpretation of these data in terms of the residual stress model.

The straightforward use of the proper experimentally derived parameters can yield a calculated composite strength which is a meaningful yardstick for evaluating performance. The utilization of rule-of-mixtures in conjunction with an arbitrary selection of the filament or matrix strength contribution makes a farce of the representation. A 400,000 psi strength filament does not become a 300,000 psi strength filament by proof stressing it to that value but it does make it easier for composite values to be represented as fulfilling the rule-of-mixtures.

The utilization of composite strain measurements in conjunction with filament modulus values to calculate filament strength utilization is based on one of the assumptions of the rule-of-mixtures ( $\varepsilon_{\rm C} = \varepsilon_{\rm f} = \varepsilon_{\rm m}$ ). The use of this assumption to imply compliance with the rule-of-mixtures is fallacious. The assumption breaks down precisely when the filaments begin to break and load is redistributed via the matrix. The load redistribution provides an additional increment of elongation. The attribution of that extra elongation to filament strain provides the appearance of extra strength utilization when multiplied by the filament modulus. This technique has been utilized to claim filament strength utilization values 20% higher than the values calculated by subtracting the matrix contribution from the composite strengths reported in the same papers.

Bundle strength concepts have been evoked to explain low composite properties relative to rule-of-mixtures calculations. Bundle strength calculations assume the independent fracture of individual filaments at their lowest strength. The utilization of bundle strength calculations for composite specimens assumes that the matrix does not act in its desired role as a load transfer medium for the accomplishment of repeated fracture of incorporated

filaments. Thus the accomplishment of bundle strength predictions in composite form is an admission of poor composite performance not the achievement of expected filament strength utilization.

Synergism is a concept which has been applied as an explanation for outstanding results when the combination of previously described misrepresentations has been particularly fortuitous. The experimental difficulties in acquiring the proper numbers for a rule-of-mixtures calculations are significant but the consequences of ignoring the importance of the right numbers has particularly clouded the interpretation of composite tensile properties.

### a. Tensile Modulus

The initial tensile modulus of filament reinforced metal matrix composites follows or exceeds the values which are predicted by the application of a simple rule-of-mixtures formula:

where 
$$E_c = V_f E_f + V_m E_m$$

$$E_c = the tensile modulus of the composite E_f = the tensile modulus of the filament E_m = the tensile modulus of the matrix  $V_f + V_m = the volume fraction of filament and matrix respectively$$$

The original verifications of the applicability of the modulus rule-of-mixtures in Cu-W composites<sup>(3)</sup> has been repeated in essentially every composite system for which viable test specimens could be fabricated. It should be emphasized that compliance with rule-of-mixtures modulus on uniaxial tensile specimens simply indicates that two materials tested in parallel will yield their proportional contribution to the test results. The compliance of experimental modulus data for a series of composite systems with rule-of-mixtures predictions is demonstrated in Figures 19a and 19b. These Data represent the initial modulus of the various systems where both the matrix and filament are being stressed elastically. A secondary modulus which can be measured after the matrix has begun to deform plastically is also reported by many investigators but interpretation is dependent upon a reliable measure of the matrix tangent modulus in that range.

The data of Herman<sup>(41)</sup> and Schmitz<sup>(42)</sup> in Figure 19c demonstrate the effect of the formation of a reaction product during the fabrication process. Schmitz's specimens were fabricated by a short time hot-pressure bonding process which minimized the formation of the boride layer on the boron filament. Herman's specimens were fabricated by a longer time diffusion bonding cycle which resulted in the formation of a larger volume fraction of reaction product. The positive deviation of modulus measurements in Herman's data can be attributed to the formation of a significant surface layer of a high modulus constituent. The fact that the deviation increases with increasing volume fraction of filament reinforces that interpretation.

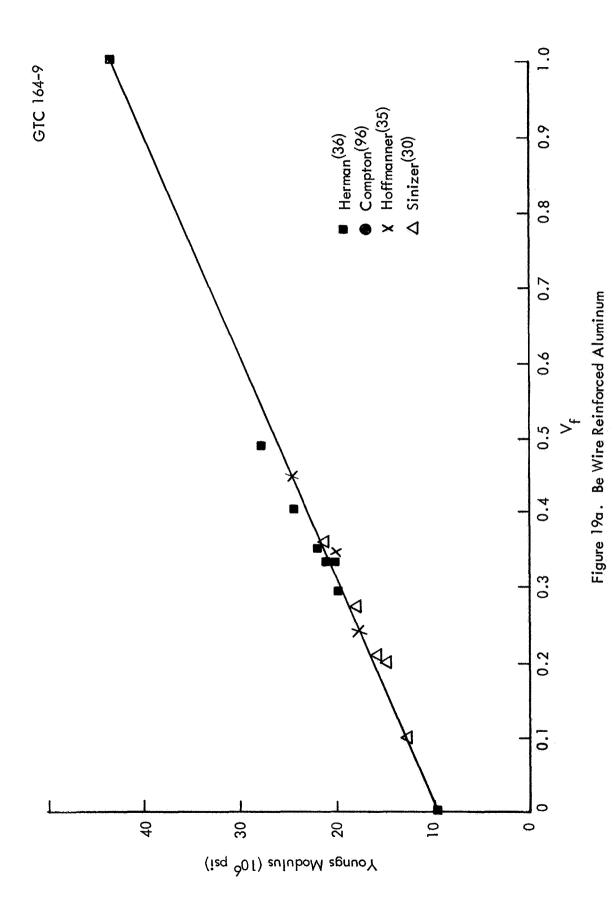


Figure 19b. Aluminum Matrix Composite Rule of Mixtures Representation of Moduli

20

(isq <sup>3</sup>01) suluboM s'gnuoY

0

0

30

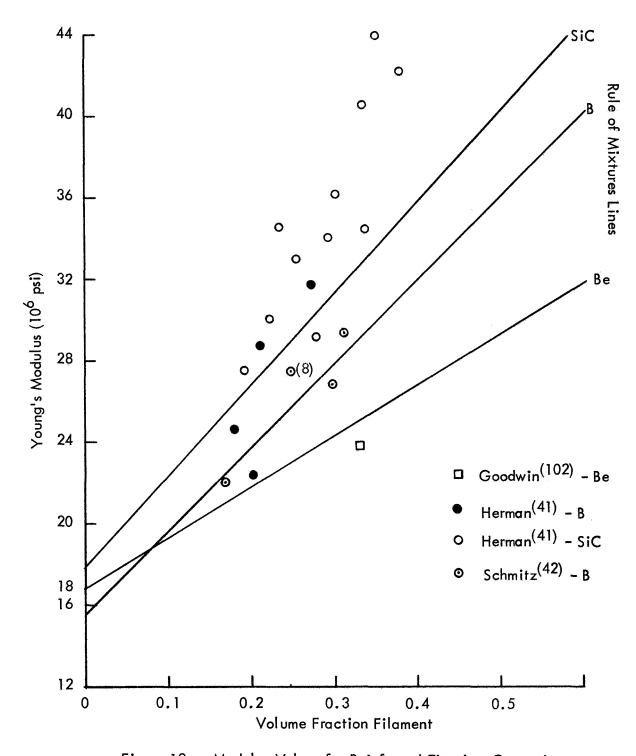


Figure 19c. Modulus Values for Reinforced Titanium Composites

### b. Tensile Strength

The data presented in Figure 20 provide the most vivid description of the state-of-the-art with regard to the tensile strength characteristics of aluminum-boron composites. Davis (31) reports the average strengths and standard deviations for his production experience on this material. Schaefer (97) utilizing quality checked material supplied by Davis has conducted the most comprehensive characterization of room temperature tensile properties in that system. Both collections of data demonstrate the increasing variability in properties and the negative deviation from a rule-of-mixtures prediction in strength with increasing volume fraction of filament. The data of Kreider (39) on AI-BORSIC are seen to fall within the envelop of Schaefer data as do the results of Young (53), Shimizu (55), Stuhrke (54) and Antony (101).

Figure 21 is a rule-of-mixtures representation of a selected group of composite samples. For these specimens the virgin filament strengths averaged 454,000 psi at a 1 inch gauge length, while the strength of filaments extracted from fabricated composites averaged 402,000 psi. This represents a fabrication degradation of only 11%. Antony<sup>(101)</sup> reports a 15% degradation in filament strength as a result of a similar fabrication procedure utilizing a somewhat higher strength filament. The experimental composite strength values extrapolate to a filament strength of 400,000 psi, indicating excellent utilization of residual filament strength.

### c. Filament Orientation Effects upon Composite Tensile Properties

The use of filament reinforced composites in sheet form requires the development of adequate biaxial mechanical properties. A large proportion of the metal matrix composites work has been conducted on uniaxially reinforced materials. The outstanding longitudinal properties of such composites were accompanied by transverse properties which were some fraction of the matrix properties (30,39,41,80,97). Two detailed studies of composite tensile properties as a function of misorientation have been conducted by Jackson (108) in the Al-SiO<sub>2</sub> and Al-stainless steel systems and by Cooper (85) for the Cu-W system. The work of Jackson indicated a surprising tolerance to low degrees of misorientation in uniaxial composites and an amazing retention of axial strength in crossplied specimens at filament orientations as high as  $\pm 20^{\circ}$ . Cooper observed a dropoff in strength for uniaxial filament reinforced specimens, Figure 22, which followed closely the theoretical prediction of Stowell and Liu (107). The room temperature tensile data for the Cu-W system are a classic example of the form of curve which has been documented in a number of composite systems. The Cu-W specimens were formed by electrodeposition and were demonstrated to have a very weak filament-matrix bond and the test specimen length was varied to prohibit the gripping of any filament at both ends.

To the contrary the aluminum matrix composites exhibited a higher degree of interfacial bonding and some filaments were continuous from grip to grip at the lower angles of misorientation ( $<15^{\rm o}$ ). In both investigations the mode of failure shifted from tensile fracture of the filaments to mixed shear and tensile failure to matrix shear failure to matrix tensile failure. The room temperature tensile results for crossplied aluminum matrix composites tested with the filament plies oriented at  $\pm \theta^{\rm o}$  to the specimen axis are

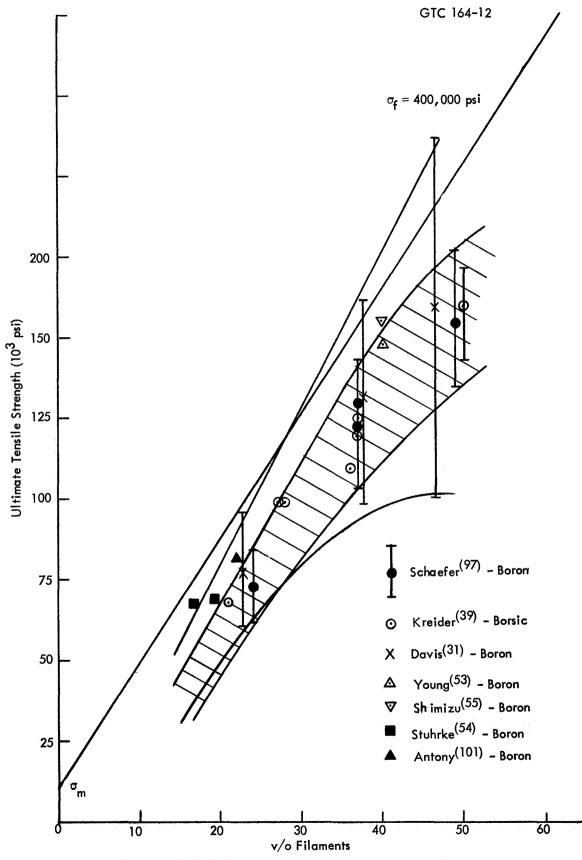


Figure 20. Tensile Strength of Al Matrix Composites as a Function of Volume Fraction Filament

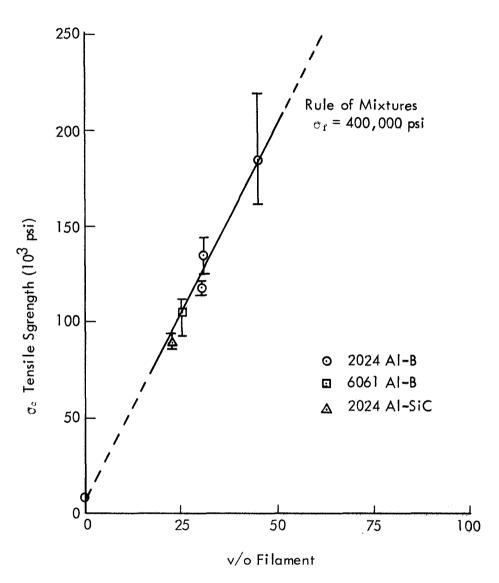
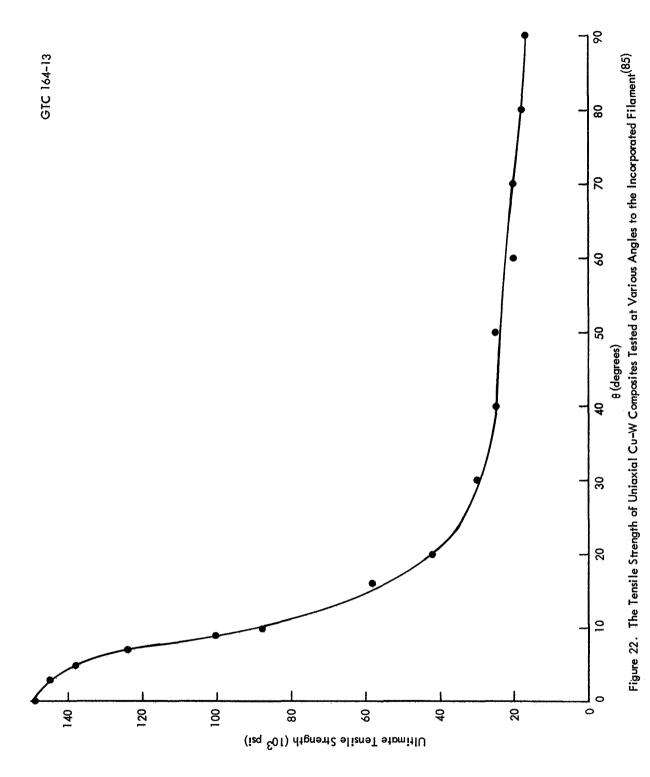


Figure 21. Range of 2024 Al Matrix Composite Properties vs Volume Percent Loading



shown in Figure 23. A number of other programs have included tensile strength results as a function of filament misorientation from the tensile axis. The expected form of off axis properties is reproduced in Figure 24 by the data of Schaefer  $^{(97)}$  and Kreider  $^{(39)}$  for 50 v/o B and 50 v/o BORSIC filament respectively. The correspondence in results is excellent. Kreider has also observed the existence of the three types of failure modes predicted by Stowell and Liu, Figure 25, where tensile failure occurs at  $^{00}$ , mixed tensile and matrix shear failure occurs at  $^{50}$  and  $^{100}$ , shear failure at  $^{200}$ ,  $^{300}$  and  $^{450}$ , and the  $^{900}$  failure appears to be related to matrix tensile failure. The data of Conliffe  $^{(80)}$  are represented in Figure 26 in comparison with the predictions of Stowell and Liu  $^{(106)}$  and Tsai  $^{(107)}$ .

The accumulated data seem to indicate that the off-axis tensile properties of boron and BORSIC reinforced aluminum drop to the range of 10,000 to 20,000 psi at test angles of 30° to 90° from the filament axis regardless of volume percent loading (Figure 27), aluminum alloy (Figure 28) or heat treated condition. However, the data of Herman on Be reinforced aluminum indicates a distinct effect of alloy matrix modification by heat treatment, Figure 29. The transverse strength of the as pressed material closely approximates the transverse strength of the matrix and the increase in matrix strength by heat treatment results of excellent load transfer capability is absent in the Al-B, Al-BORSIC systems of Al-stainless steel systems. Similarly the Al-Be composite system shows excellent biaxial properties in orthogonal crossply panels compared with Al-B composites tested at various angles to the principal axes, Figure 30. The sufficiency of the interfacial bond between filament and matrix is evidently involved in this difference in behavior. The examination and identification of the origins of the differences could lead to better biaxial properties in oriented crossply composites of the other filament reinforcements for the aluminum matrix.

Figure 31 compares uniaxial Al-50 v/o B composite properties as a function of orientation with a 0-90° crossply and a  $\pm$  30° crossply (97). The ability to tailor biaxial properties is readily apparent but so is the price which must be paid in absolute strength to accomplish any required level of off-axis properties. Off-axis tests on unidirectional composites show a significant decrease in modulus as indicated in Figure 32 but the 0-90° crossply and the  $\pm$ 30° crossply specimens yield almost constant modulus values.

The data of Herman<sup>(36)</sup> on beryllium wire reinforced aluminum composites again show the advantage of excellent filament matrix bonding in maintaining off-axis mechanical properties, Figure 33. The experimental elastic modulus values fall significantly above solid line which represents the Tsai analysis<sup>(112)</sup> based on Haskin's<sup>(113)</sup> parallel cylindrical inclusion theory for the transverse modulus. The ability of the filament matrix interface to support the added stresses which are imposed by the tendency of the specimen to shift laterally between rigid testing grips undoubtedly contributes to these apparently high values.

# d. Strain Rate Effects Upon Ultimate Tensile Strength and Modulus

Kreider (39) observed no effect of strain rate variations upon the ultimate tensile strength of 50 v/o BORSIC reinforced aluminum composites in the range from 0.1 to

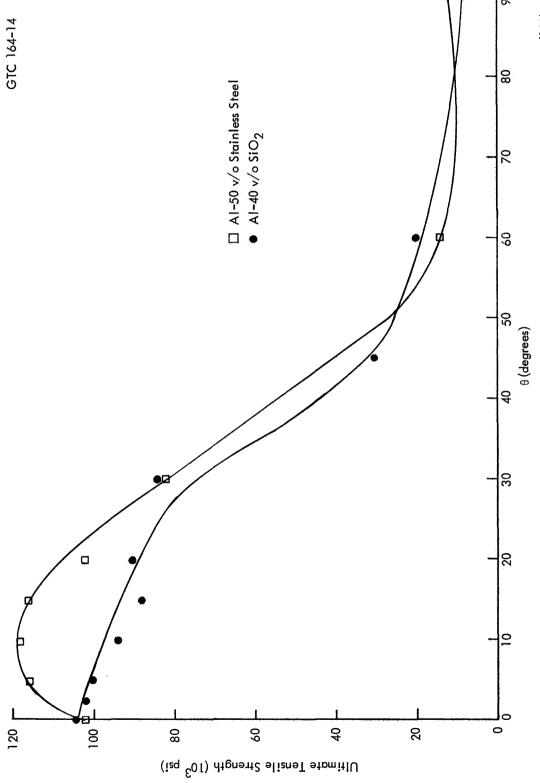


Figure 23. Tensile Strength Results on Laminated Crossply Panels with Filaments Oriented  $\pm~\theta$  to the Specimen Axis $^{(108)}$ 

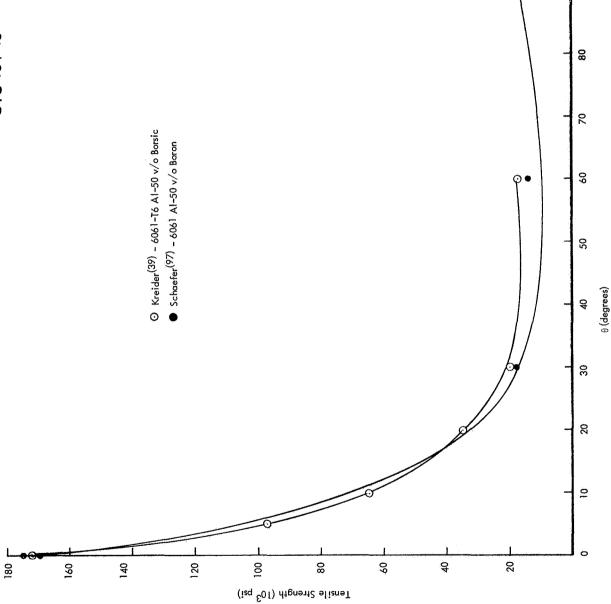


Figure 24. Uniaxial Composite Tensile Properties as a Function of the Angle Between the Fiber Axis and the Tensile Axis

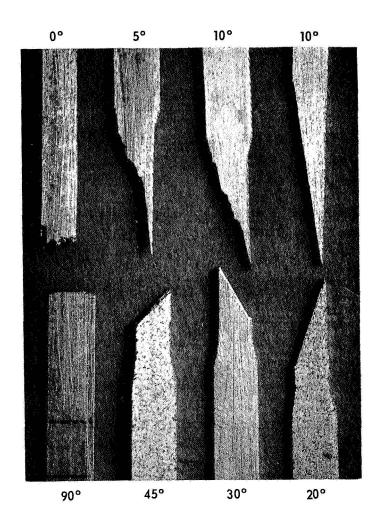


Figure 25. Fractured Surfaces of Borsic-Aluminum Composites.

Specimens Were Tested with Given Angle
Between Fiber Axis and Tensile Axis

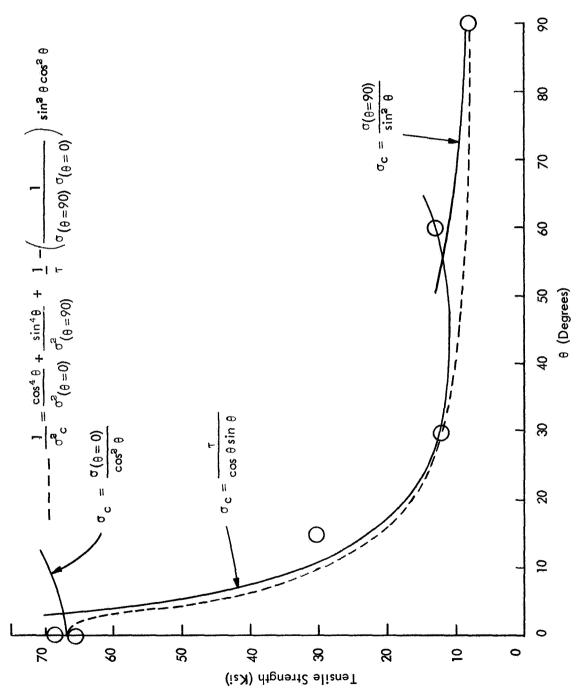


Figure 26. Tensile Strength Variation with the Angle Between Uniaxially Oriented Filament and the Tensile Axis

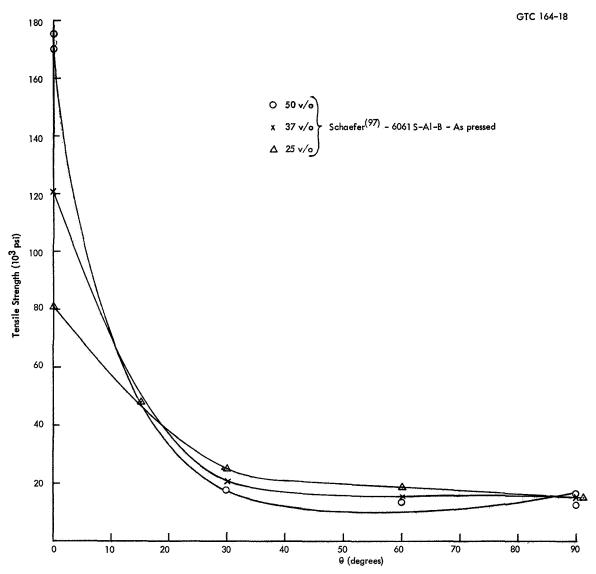


Figure 27. Effect of v/o Filament on Off-Axis Tensile Properties

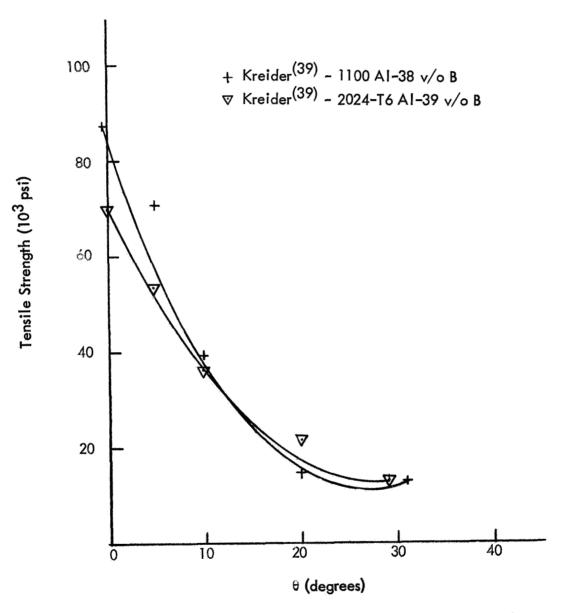


Figure 28. Effect of Aluminum Alloy on Off-Axis Tensile Properties of Boron Reinforced Composites

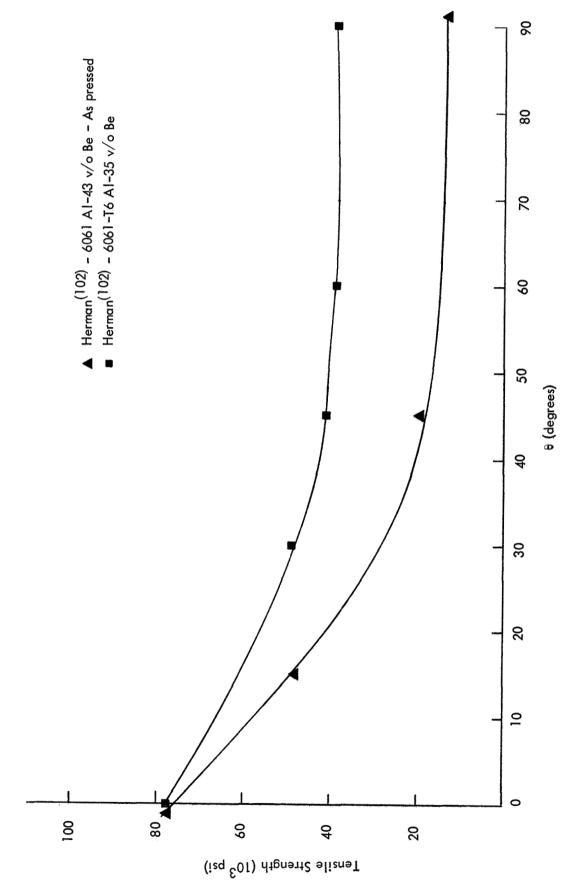


Figure 29. Effect of Aluminum Alloy Heat Treatment upon the Off-Axis Tensile Properties of Be Reinforced Composites

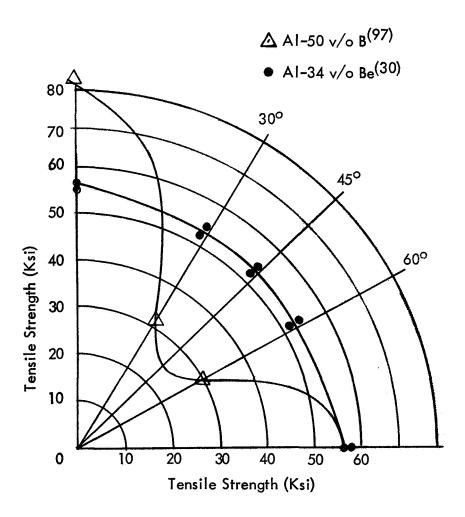


Figure 30. Off-Axis Properties of Al-34 v/o Be and Al-50 v/o B Othogonal Crossply Composites

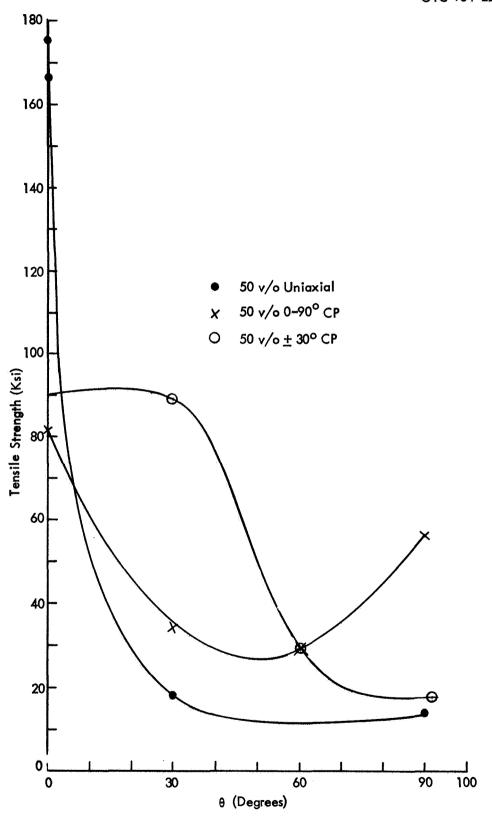


Figure 31. Comparison of Off-Axis Properties of Uniaxial 50 v/o Boron Filament Reinforced Aluminum with 0-90° and  $\pm$  30° Crossplys (97)

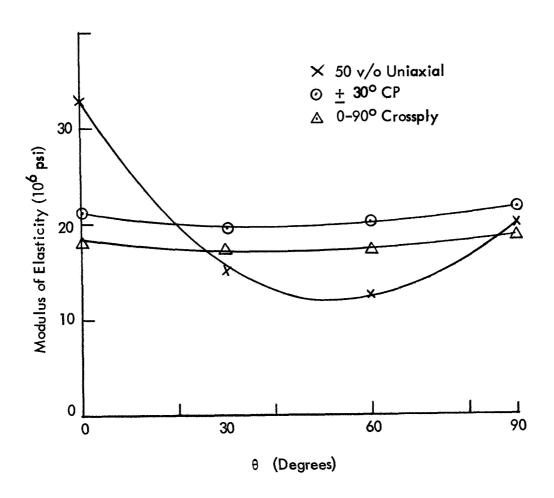


Figure 32. Modulus of Elasticity Versus Orientation for 50 v/o Uniaxial, 0-90°Crossply and ± 30° Crossply Aluminum-Boron Composites

GTC 164-24

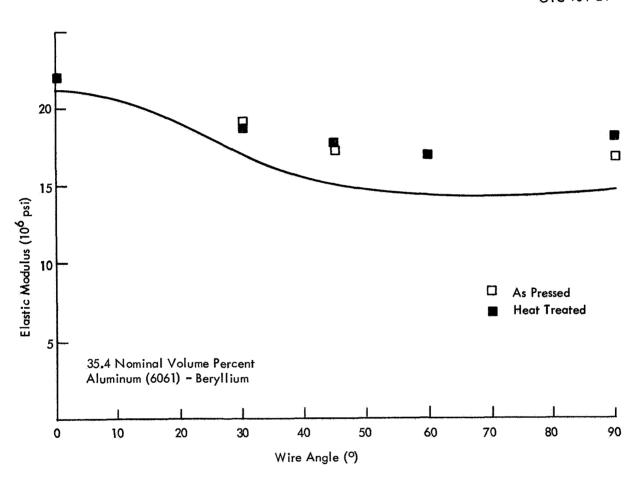


Figure 33. Modulus of Elasticity Versus Wire Orientation

1.0 in/in/min, Figure 34. Lenoe<sup>(111)</sup> covered a considerably wider strain rate range from .005 to 905 in/in/min on 65 v/o boron reinforced melt infiltration specimens and also observed no strain rate effect upon strength or modulus, Figure 35. These were somewhat surprising results in that some time dependence might be expected in the stress redistribution matrix shear process which occurs on testing composites to failure.

#### e. Notch Sensitivity

Notch sensitivity in filament reinforced metal matrix composites might be expected to be low since individual fiber fractures are likely to be more severe stress concentrators than are machined notches. Indeed Kreider (32) observed no significant notch effect in plasma sprayed and hot pressed boron-aluminum composites with stress concentration factors of 2.9. However, the data of Schaefer (97) show a distinct notch sensitivity in 0.020 inch thick 25 and 50 v/o unidirectional boron reinforced aluminum composites at a notch acuity of approximately 6.3. Notched/unnotched strength ratios of .71 and .82 were reported for the 25 v/o and 50 v/o samples respectively. To the contrary the notched strength of the 0.080 inch thick specimens with the same filament loading were only 5% lower than the unnotched results on specimens from the same composite panel. The same pattern was exhibited for the results on thick and thin specimens of 50 v/o orthogonal crossplied material. Since notch insensitivity for filament reinforced composite materials has been a sort of intuitive assumption, the origins of discrepancies such as these need to be examined in detail. The analysis of notch effects in homogeneous materials is difficult and interpretation of generated results are rife with specimen size and scale effects. The introduction of triaxiality by mechanical means upon a material with a self-generated gradient of triaxiality can be expected to provide the basis for more than a casual examination of composite toughness and fracture mechanics for the future.

#### f. Poisson's Ratio

The experimental measurements of Poisson's ratio for metal matrix composites are sparse and scattered. Strain gauge rosette techniques were applied to tensile test specimens to evolve  $\varepsilon_0$  and  $\varepsilon_{90}$  values in the aluminum boron system by both Kreider (39) and Young (53). Their results for various volume percent loadings and for various filament orientations are presented in Table IV.

Table IV Poisson's Ratio Values for Various Al-B Composites

Filament Value Fraction	Orientation	Poisson's Ratio	Reference
.53	uniaxial	. 219	(39)
.48	uniaxial	. 227	(39)
.42	uniaxial	.309	(53)
.42	± 22.5°	.374	(53)
.42	± 45°	.318	(53)

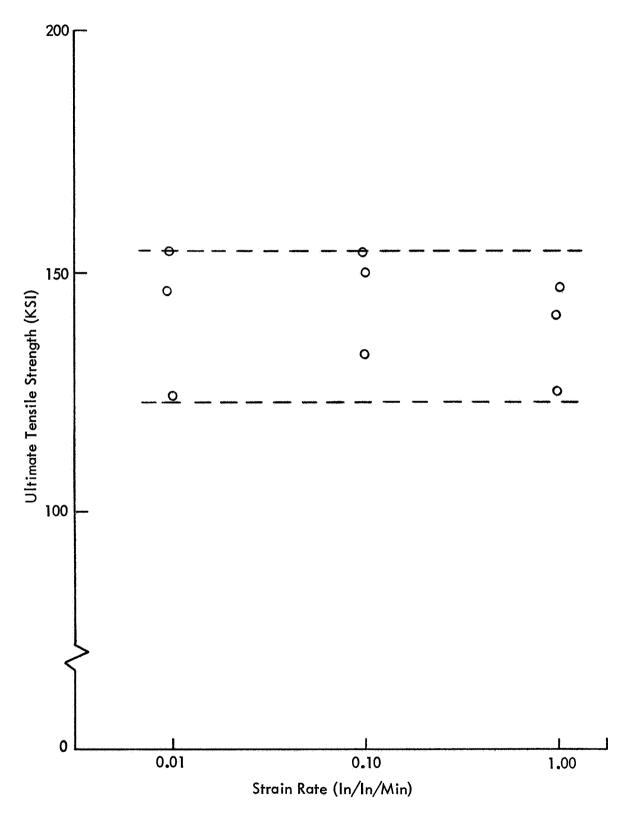


Figure 34. Effect of Strain Rate on Ultimate Tensile Strength Borsic–Aluminum Composites (Nominal Fiber Content 50%)

8 T

(izq <sup>8</sup>01) suluboM S

20

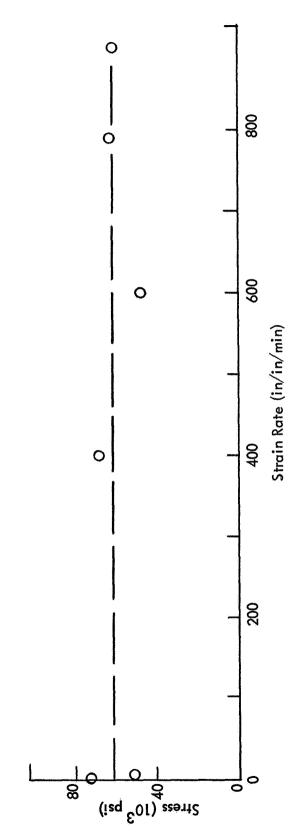


Figure 35. Effect of Strain Rate on Modulus and Tensile Strength of Al–65 m v/o~B~Composites

Similarly Baker  $^{(89)}$  has reported Poisson's ratio values ranging between .22 and .29 for  $50 \text{ v/o SiO}_2$  reinforced Al specimens at low and high strains respectively.

### 2. ELEVATED TEMPERATURE PROPERTIES OF COMPOSITES

The potential for improved retention of room temperature strength to higher applicational temperatures has been the basis for much of the developmental interest in composite concepts. The experimental effort has been concentrated on characterizing the elevated temperature behavior of specific composite combinations as opposed to studying short-time tensile fracture, stress-rupture or creep processes. Short-time elevated temperature tensile properties have been shown to follow the simple rule-of-mixtures predictions which are applicable at room temperature. Composites creep occurs at the very low rates dictated by the incorporated filament and creep rupture life is considerably extended. The interpretation of the meager data which are available is quite speculative, though the framework for thinking about such properties has been developed by Kelly (28) in a model which was consistent with his observations and by de Silva (105) who has presented a phenomenological analysis of creep in fiber reinforced composites. McDanels (104) has developed equations for the prediction of creep and stress rupture properties from the respective properties of composite constituents. These references serve as a basis for discussing experimental observations but tests of their validity have yet to be undertaken.

## a. Short-Time Elevated Temperature Tensile Properties

The short-time elevated temperature tensile tests performed on aluminum matrix composites by a number of investigators are presented in Figure 36. It is clearly demonstrated that strength begins to drop off rapidly between 500 to 600°F. Data in the range from room temperature to 500°F may show some vacillation in individual programs or a small maximum which can easily be attributed to the effects of varying times at those temperatures upon matrix properties and to modification in the residual states of stress in the composite. Variability seems lower in most instances at elevated temperature than at ambient. The work of Kreider (39) is sufficiently comprehensive to permit the calculation of standard deviations on the strength af four temperatures. The data of Schaefer (97) and Young<sup>(53)</sup> at the same strength level fall within the envelope of the Kreider data and that of Cratchley<sup>(91)</sup>, Taylor<sup>(100)</sup>, Compton<sup>(96)</sup> and Antony<sup>(101)</sup> take on the same form at different strength levels. The similarities in form are notable considering that four different types of reinforcement are represented. The drop-off in short-time tensile properties between 500°F and 600°F coincides with a drop-off in matrix strength in the same temperature range. It is apparent that as the matrix is drastically weakened with increasing temperatures, its contribution to composite strength and its ability to redistribute load upon the initiation of filament fracture is reduced. Thus the composite samples for each reinforcement regardless of strength level exhibit the rapid decrease in tensile strength as the matrix is weakened. It is clear from a multitude of digestion experiments that filament strength degradation does not occur as a result of short-time exposures to temperatures under 930°F. Figure 37 reflects the same phenomenon as measured by Baskey (69) for tungsten wire reinforced superalloy composites. Figure 38 illustrates the characteristic form of elevated temperature tensile data for representative aluminum, titanium and nickel matrix composites.

X Schaefer<sup>(97)</sup> - Boron

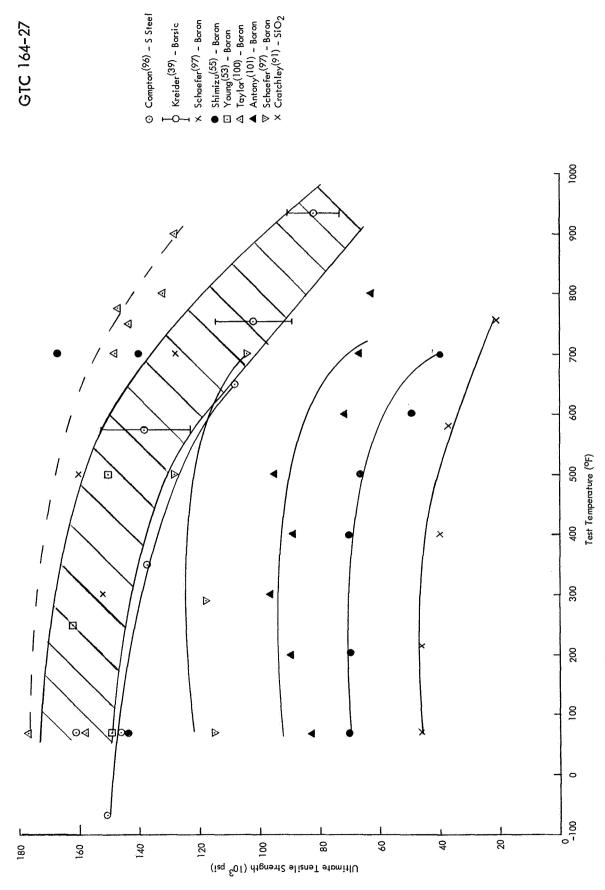


Figure 36. Tensile Strength of Filament Reinforced Aluminum Composites as a Function of Temperature

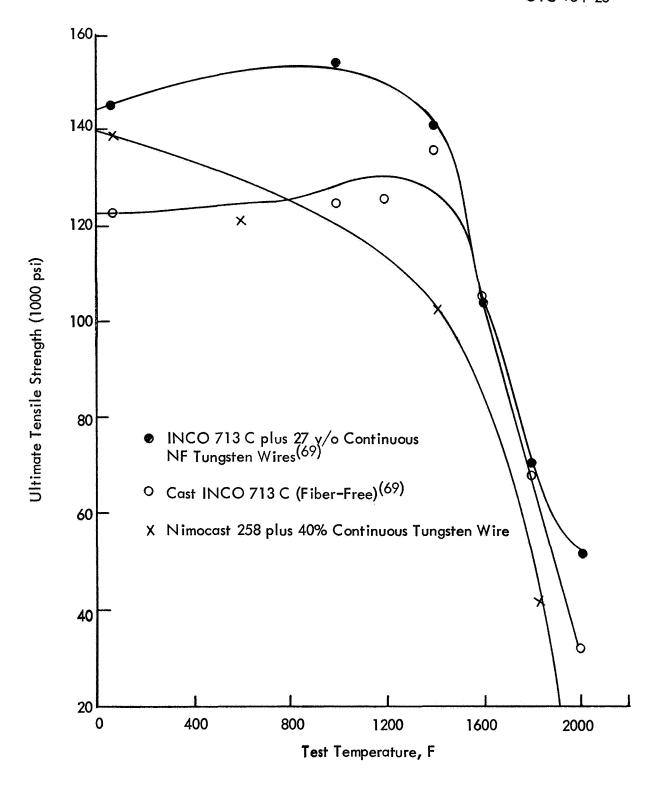


Figure 37. The Elevated Temperature Tensile Strength of Tungsten Reinforced Nickel Alloys as Compared to Matrix Properties.

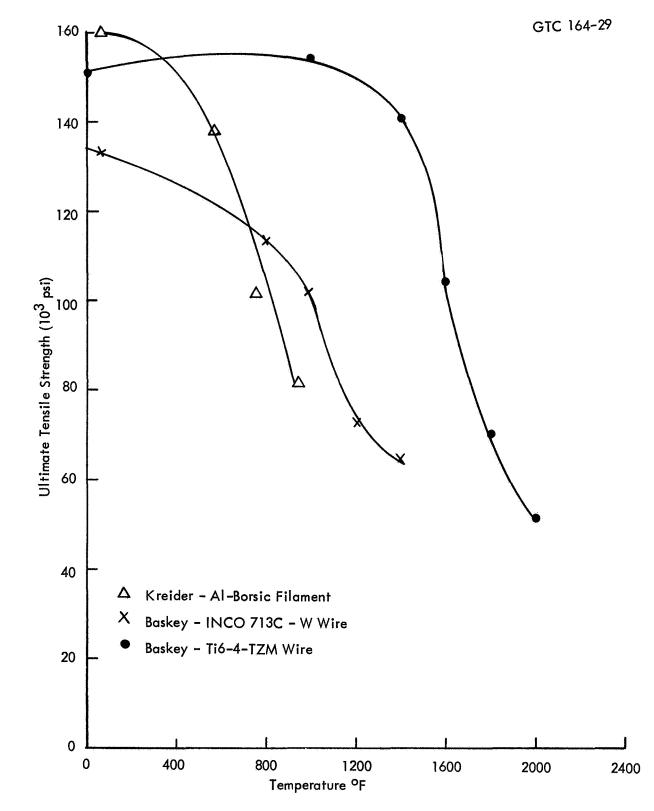


Figure 38. The Effect of Temperature upon the Tensile Strength of A Representative Composite in the AI, Ti and Ni Matrices

# b. Elevated Temperature Elastic Modulus

The decrease in elastic modulus with increasing temperature is demonstrated by Baskey (69), Figure 39, for nickel and titanium matrix composites reinforced with refractory metal wires. Similarly Young (53) and Compton (96) have reported decreases for boron and stainless steel reinforced aluminum composites, Figure 40. An unexplained increase in modulus at 300°F which is evident from the data of Schaefer, Figure 41 should be investigated. The development of a particularly effective filament matrix bond and the relaxation of residual tensile stresses in the matrix might contribute to such a rise. The decrease in matrix and filament modulus with increasing temperature would be expected to yield the type of decrease reflected in Figures 39 and 40.

## c. Creep and Stress Rupture

The creep and stress-rupture properties of filament reinforced composites can be considered to represent complexity squared. The phenomena are complex and the material is complex. The exposure of non-equilibrium combinations of filament and matrix to elevated temperatures can result in interactions which may be beneficial or degrading to either phase. The relaxation of residual stresses can modify the resultant properties and the individual deformation and fracture behavior of either constituent can be controlling in the creep or ultimate rupture process for the composite. The complexities of creep and stress rupture in composite materials have not been studied but the properties have been characterized by a number of investigators(30,39,53,56,69,80,83,88,96,97,101,116,117). The general conclusion is that extremely low composite creep rates are observed and stress rupture life is considerably extended relative to the properties of the matrix. Creep rates appear to be controlled by the incorporated filament creep rates and ultimate fracture occurs by the accumulation of filament fractures which shifts control to the deformation characteristics of the matrix.

de Silva<sup>(105)</sup> has presented a phenomenological analysis of creep in fiber reinforced composites which differs from the model proposed earlier by Kelly and Tyson<sup>(116)</sup> to explain their experimental creep observations. A concerted effort should be undertaken to experimentally evaluate the framework which is provided by these papers utilizing a composite system which is sufficiently advanced to yield consistent results. Similarly McDanels<sup>(104)</sup> has undertaken to develop predictive relationships for the creep and stress rupture properties of fiber reinforced composites which are essential to the engineering design of materials for high temperature application. The validity of these relationships when applied to the model copper-tungsten system and to the nickel-tungsten was demonstrated.

Attempts to measure the creep curves of composite samples have resulted principally in the measurement of grip slippage and/or loading train elongations far in excess of the actual deformation of the sample. Conliffe  $^{(80)}$  measured creep rates in the  $10^{-4}$  in/in/hr range for 30 to 50 v/o B-Al composites at  $500^{\circ}$ F but considered this to be a very conservative estimate. Kreider  $^{(39)}$  emphasized the difficulty in measuring specimen plastic elongation in such materials. Measured values of  $10^{-5}$  in/in/hr at  $400^{\circ}$ C and  $10^{-3}$  in/in/hr at  $500^{\circ}$ C on 50 v/o filament reinforced aluminum are consistent with the observation

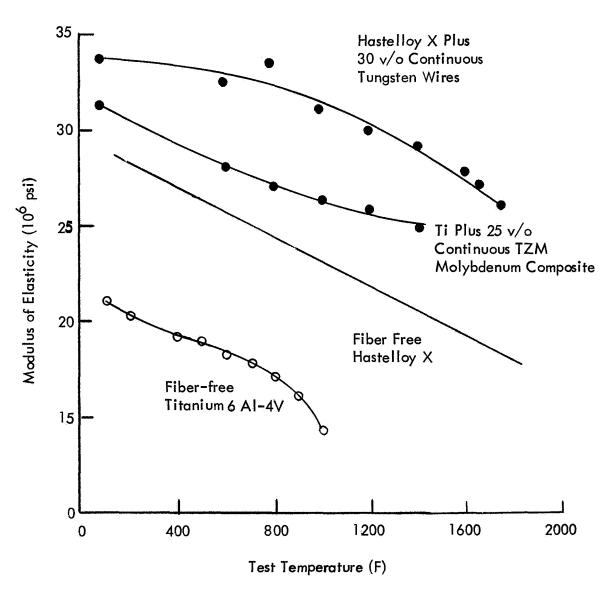
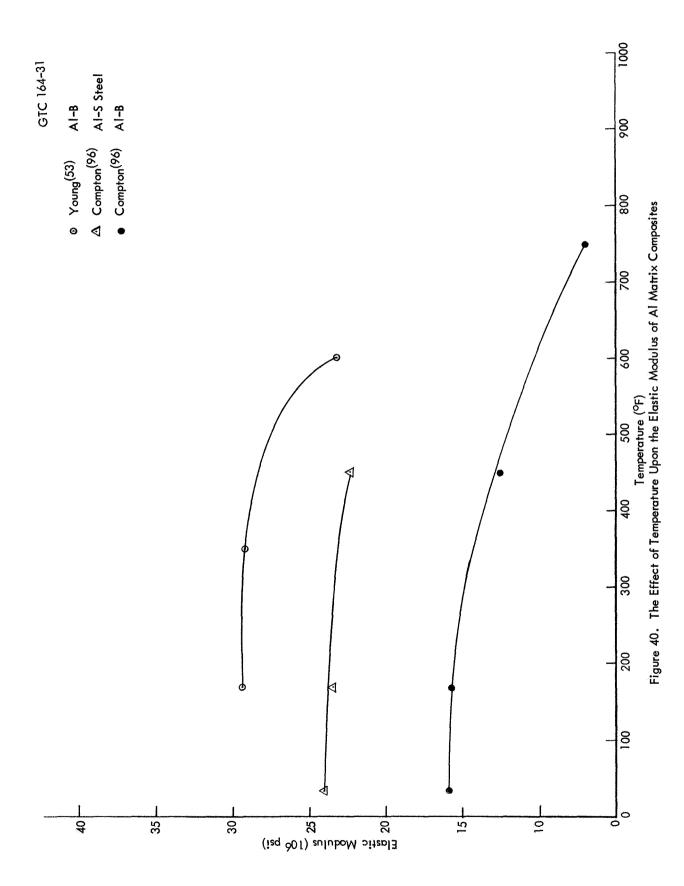
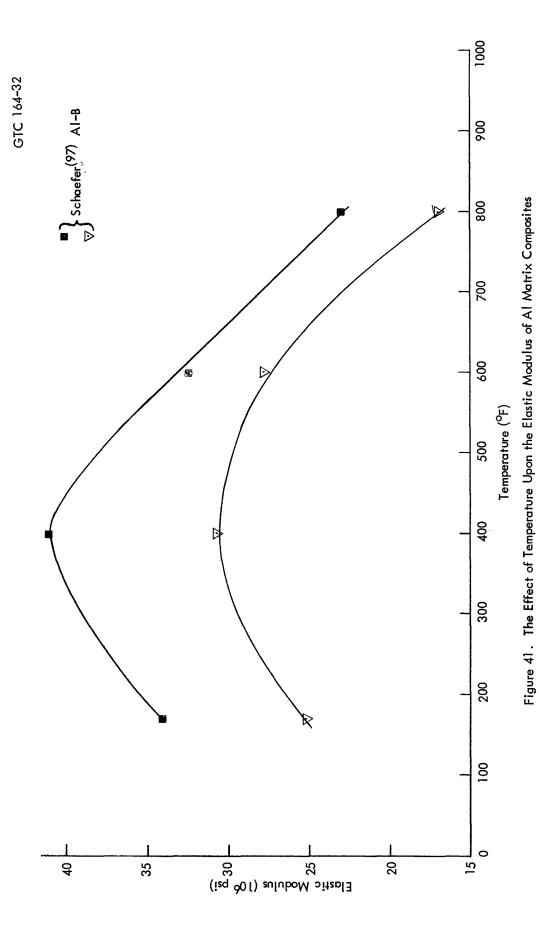


Figure 39. Effect of Temperature on Nickel Matrix and Titanium Composite Modulus of Elasticity





that boron filament exhibits creep rates below  $5 \times 10^{-5}$  in/in/hr at  $1200^{\circ}$ F under a 200,000 psi load. Composite specimens that had been tested to rupture were examined by dissolution of the matrix for separation of fractured fiber ends. Very few such separations were observed while the filaments were held in place by the matrix, however, many breaks were evident when the matrix was completely dissolved. The cumulative contribution of the breaks could not be quantitatively evaluated. However, the absence of gross open fiber breaks indicate that substantial plastic elongations were not taking place. Baker (110) working with  $50 \text{ v/o SiO}_2$ -Al composites indicated the lack of measurable creep after 100 hours at  $350-450^{\circ}$ C at stress levels as high as 40,000 psi, Figure 42. Miura (117) reported extremely low strain rates of  $10^{-6} - 10^{-7}$  in/in/hr for 20 v/o W filament reinforced aluminum at  $350^{\circ}$ C under a load of 8600 psi.

Creep curves for the metal fiber reinforced system Ni-W have been reported by Baskey<sup>(69)</sup>. Continuous filament composites were shown to yield guite low creep rates relative to the properties of the matrix, Figure 43. However, the creep rate at 2000°F was observed to be considerably greater than would be predicted by rule-of-mixtures calculations based on the behavior of the composite constituents. The development of triaxial conditions of stressing due to Poisson's ratio differences between the filament and matrix may account for the apparent increase in creep rate. The work of Miura<sup>(117)</sup> and Kelly<sup>(116)</sup> clearly indicated that the creep rate of continuous filament reinforced composites is considerably lower than for discontinuous filament reinforced samples in the same volume percent range.

The stress rupture properties of metal matrix composites have been measured to a greater degree than the creep characteristics primarily because of the ease of generating rupture life data on test specimens and the relative difficulty of achieving reliable microstrain measurements. An extensive base of reliable high temperature strength data has been accumulated for conventional alloys utilized in elevated temperature applications. The utility of those emperical data are substantially enhanced by a sound understanding of the structural changes which occur in the material during the conduct of the tests. Changes in the mode of fracture, recrystallization, grain growth, spheriodication or oxidation are only a few of the active processes which can be clearly identified as influencing stress rupture life. Interpretation of stress rupture life in filament reinforced composite materials is complicated by the introduction of a load carrying non-equilibrium phase and the permeations and combinations of interaction which can occur as a result of long-term exposure to elevated temperatures under stress.

The stress rupture process in metal matrix composites has not been studied but characterization data indicate excellent performance when compared with conventional alloys. Below the temperature at which filament-matrix interaction begins, Baker has observed no perceptable time dependent failure process. A critical stress is necessary to cause instantaneous failure at a given temperature and below this stress the Al-SiO<sub>2</sub> system shows no marked tendency to failure even after 1000 hours. All of the experimental work on the Al-B or Al-BORSIC systems has been conducted at temperatures well below that required to accomplish gross reaction. These composites carry a high fraction of their room temperature strength for as long as several hundred hours over a temperature range from 500°F to 932°F. While the number of specimens tested in any program is small and

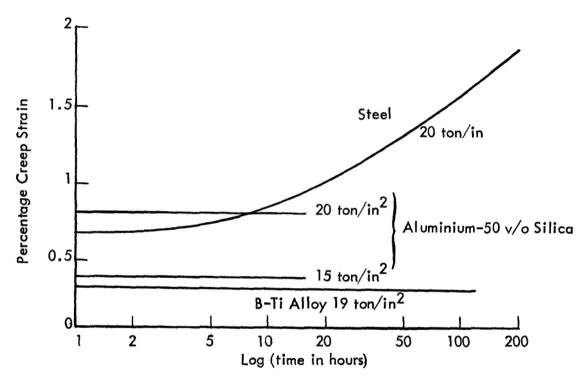


Figure 42. Creep Properties of Al-SiO<sub>2</sub> Composites and Other Materials at 400°C

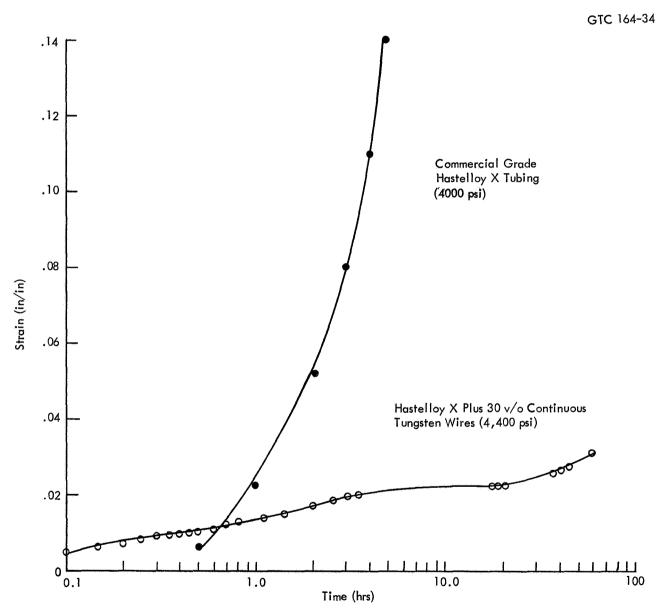


Figure 43. Creep of Hastelloy X Composite and Commercial Grade Hastelloy  $\boldsymbol{X}$ 

specimen to specimen variability is large, the idea of a critical stress below which failure does not occur would seem to better characterize the stress-rupture performance than would the idea of strong time dependent degradation in strength. Such a view of these materials is consistent with the fact that the filaments are the principal load carrying members and control the creep deformation of the composite. The data of Kreider<sup>(39)</sup> indicate the effect of temperature upon the measured creep rupture life of Al-BORSIC and Al-B composites formed by plasma spraying and hot pressing, Figure 44.

The effect of composite specimen variability upon the stress rupture curves is to lower the slope of the curve by providing short-time failures at lower stresses than are characteristic of the average strength of short-time tensile specimens. This is clearly evident from the comparison of the 0.1 hour stress rupture values with the short-time tensile strength, Table V.

Table V Comparison of Al-50% BORSIC Composite Short-Time Tensile with 0.1 Hour Stress Rupture Strengths

Temperature	Short Time Tensile Strength (10 <sup>3</sup> psi)	0.1 Hour Stress Rupture Strength (10 <sup>3</sup> psi)
300°C	138	101
400°C	102	90
500°C	82	80

The difference in values is the greatest for the lower temperature tests and least at the highest temperature where specimen to specimen variability was observed to be least.

The 500°C stress rupture properties of a 50 v/o BORSIC-Al composite are replotted in Figure 45 as compared with the conventional Ti-6Al-4V alloy. The extraordinary long-term stability at high stress levels is undoubtedly one of the most attractive characteristics of aluminum matrix composites. Figure 46 presents representative boron and stainless steel wire reinforced aluminum composite data as compared with the properties of the matrix to reiterate the magnitude of the improvement which accompanies the reinforcing fibers.

A point of interest is the greater degree of consistency and the steeper slope of the data for the ductile metal filament reinforcement as compared to the boron composites. Similarly the strain to failure is greater for the metal fiber reinforced material.

The metal fiber reinforced systems of tungsten and molydenum in nickel and copper have been exposed to extensive stress rupture testing by Baskey<sup>(69)</sup>, Dean<sup>(56)</sup>, McDanels<sup>(104)</sup> and Petrasek<sup>(118)</sup>. The data presented in Figure 47 demonstrate the almost phenomenal correspondence between the results of three investigators working on the Ni-W system and also illustrates the improvement which can be accomplished in 100 hour rupture stress by increasing the volume fraction of W. A typical stress rupture plot for tungsten reinforced nickel base alloys is presented in Figure 48<sup>(69)</sup>. The advantages of

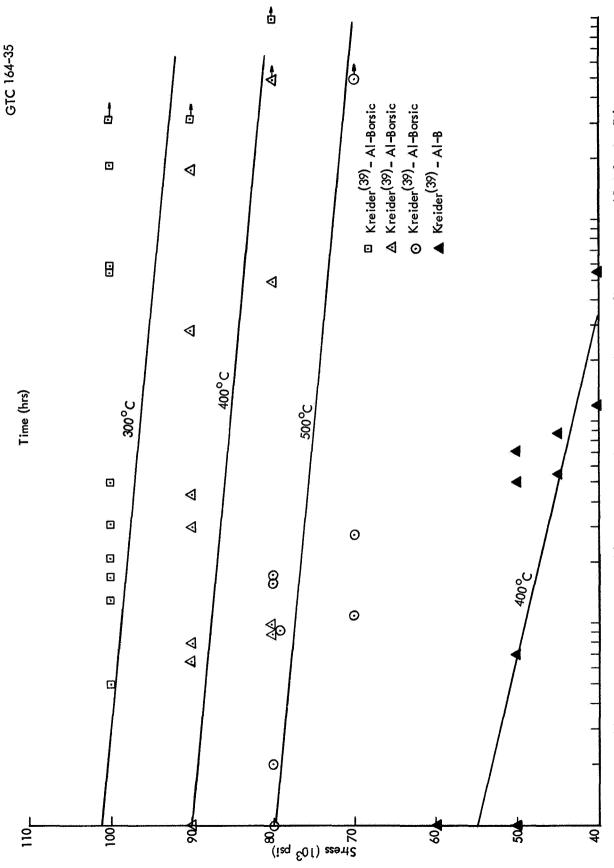


Figure 44. The Stress Rupture Life of Aluminum Matrix Composites as a Function of Temperature and Reinforcing Filament

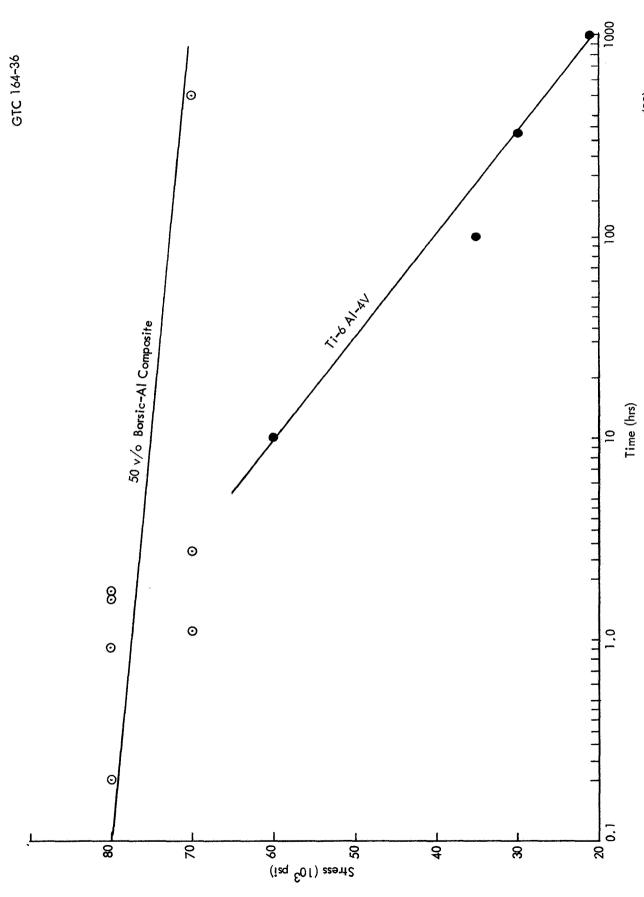


Figure 45. Comparison of 500°C Stress Rupture Life of A1-50 v/o Borsic Composite with Titanium-6 A1-4V Alloy (39)

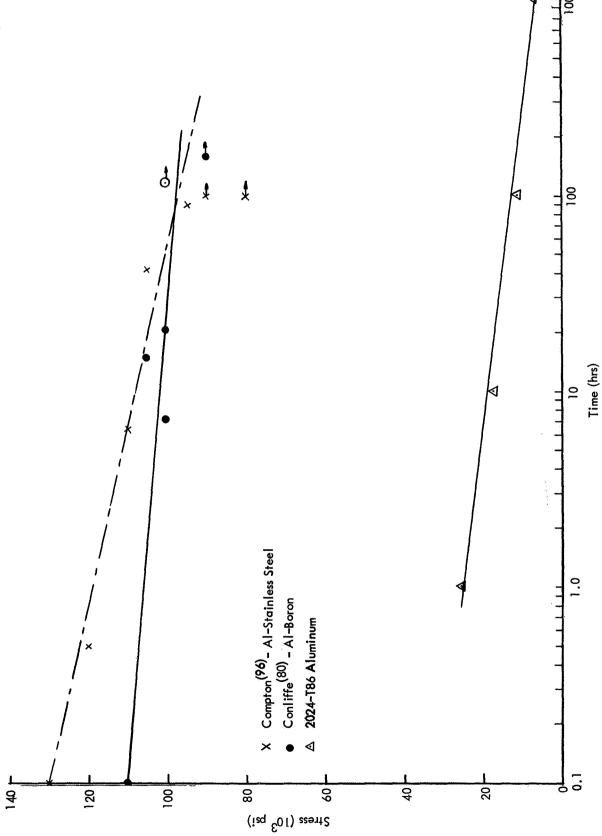


Figure 46. Comparison of the 500°F Stress Rupture Properties of Boron and Stainless Steel Reinforced Composites with the Properties of the Matrix

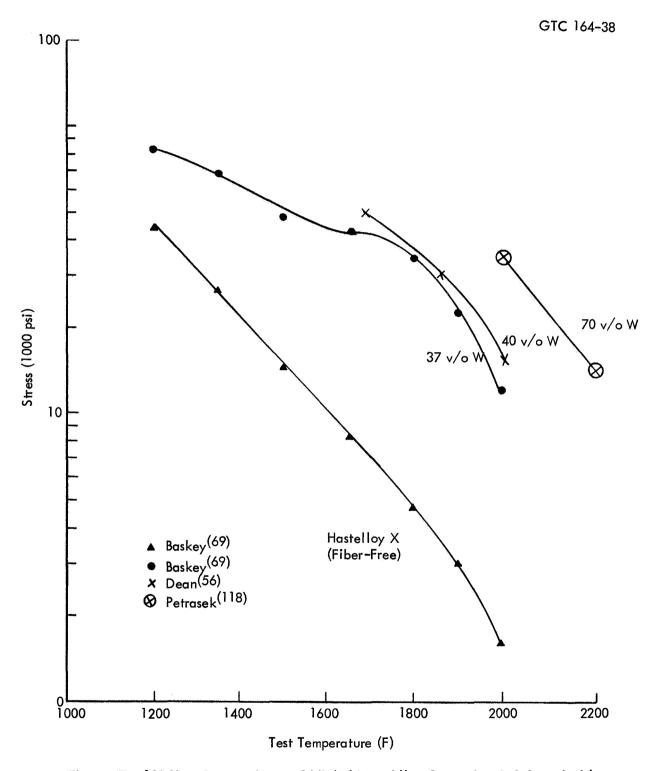


Figure 47. 100 Hour Rupture Stress of Nickel Base Alloy Composites Reinforced with Tungsten Wires as Compared with Fiber-Free Hastelloy X

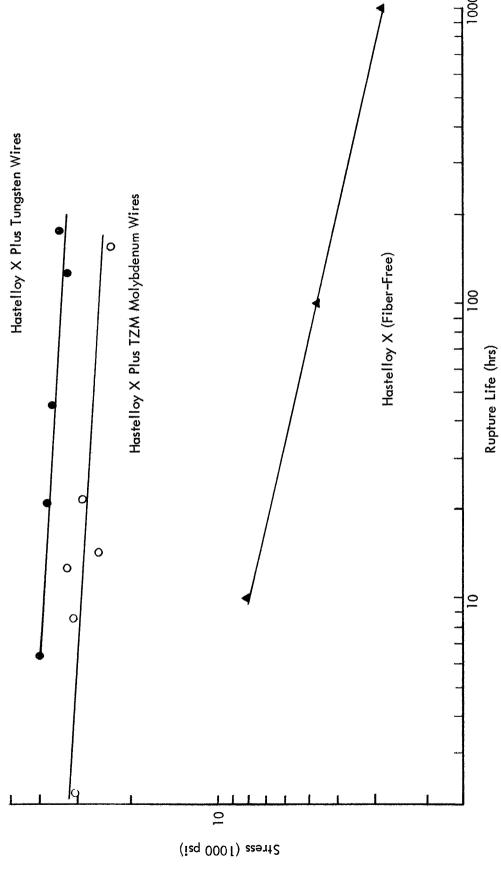


Figure 48. Stress Rupture of Hastelloy X Plus 37 v/o Continuous Tungsten or TZM Molybdenum Wires and Fiber-Free Hastelloy X at 1800 F

80

utilizing a composite system which can be fabricated to yield consistent specimens is evident from the limited scatter in experimental results. The change in stress rupture properties as a function of temperature as represented in Figure 49 indicates a change in controlling fracture mechanism. At 1900°F the slope of the composite data is less than that of the matrix while at 2000°F the composite slope is greater than the fiber-free matrix. Thus in this composite system the effect of fiber-matrix interaction is visible in elevated temperature stress rupture properties.

## d. Thermal Shock or Cycling Effects

Antony<sup>(101)</sup> conducted an illuminating series of experiments on the effect of multiple thermal shock cycles from room temperature to  $800^{\circ}$ F upon the strength of Al-27 v/o B composite specimens and upon the strength of extracted boron filaments, Figure 50. The composite strength decrease of 40% after 100 cycles was adequately accounted for by the observed 20% degradation in boron filament strength. Conliffe<sup>(80)</sup>, in a less severe series of thermal cycling experiments, 100 cycles  $100^{\circ}$ F to  $500^{\circ}$ F, could observe no significant difference in the room temperature strength of specimens in the as-received or thermally cycled condition.

In addition to thermal cycling effects upon the strength of the composites by filament degradation it should be expected that interfacial fatigue resulting from differences in constituent thermal expansion coefficients could result in a progressive loss of filament-matrix integrity with continued cycling. Similarly coated filaments, BORSIC or nitrided boron, might experience delamination or surface layer cracking which could contribute to accelerated degradation in composite mechanical properties.

#### e. Elevated Temperature Exposure

The effect of elevated temperature exposure on the room temperature tensile properties of composites was an early technique for evaluation of filament matrix compatibility (30,120,122). Toy (30) reported full filament strength retention in Al-Be composites exposed to 600°F for 1000 hours and Conliffe (80) reported no composite strength degradation in Al-B composites after 100 hours at 500°F. Alexander (120) documented the drastic effect of elevated temperature exposure upon the properties of the Ni-B composite system as filament-matrix interaction is initiated. Kreider (39) provided vivid evidence of the effect of elevated temperature exposure of boron and BORSIC filament on contact with 2024 Al, Figure 51. A drastic dropoff in boron strength was recorded after 500 hours at 200°C while BORSIC retained its strength even after 500 hours at 600°C. An additional 500 hours at 200°C had little effect on the boron while an additional 500 hours at 600°C reduced the BORSIC strength by roughly 35%. It is emphasized that the reaction degradation recorded for boron filament in Kreider's work is undoubtedly influenced by the high oxygen content of the plasma sprayed matrix and should not be considered to be representative of boron-aluminum composites fabrication by other techniques.

In the nickel-boron composite system fabricated by electrodeposition the effect of exposure to slightly elevated temperatures was observed to result in an increase in composite strength (121). As the temperature was increased the strength went through a

Figure 49. Stress Rupture of Hastelloy X Plus 36 v/o Continuous Tungsten Wires and Fiber-Free Hastelloy X at 1900 and 2000 F

82

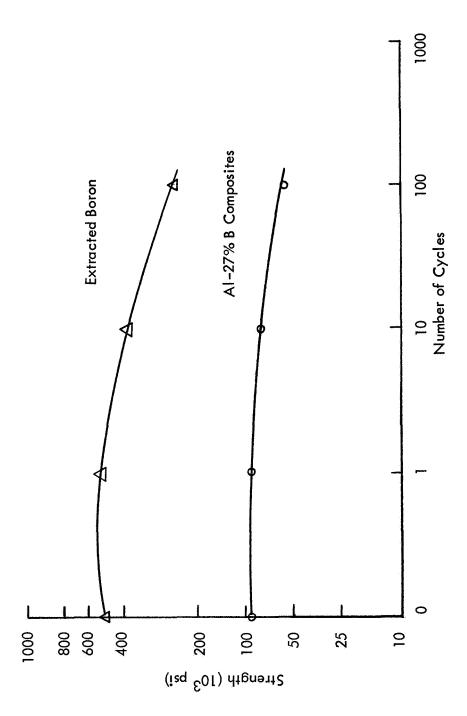


Figure 50. Effect of 800°F Thermal Shock on Tensile Strength of AI-27% B Composites



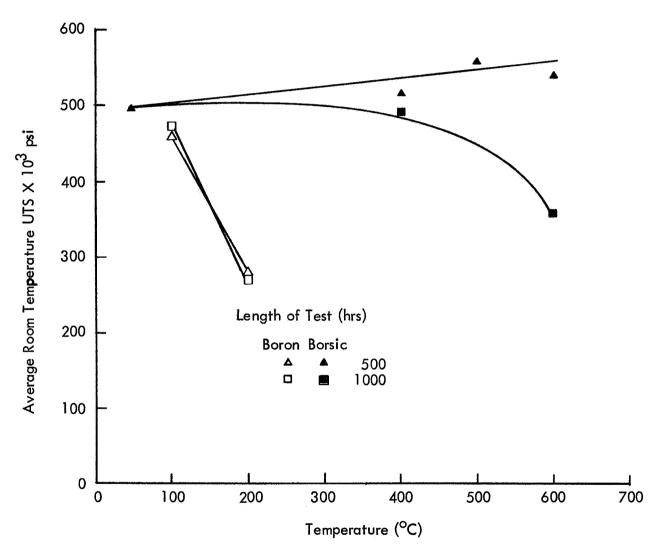


Figure 51. Room Temperature Strength of Boron and Borsic Fibers Heated in 2024 Aluminum

maximum and then degraded drastically. Such evidence of elevated temperature enhancement of composite strength has been reported in a number of systems and is attributed to the development of an improved interfacial bond prior to reaction degradation of the filament.

## 3. DYNAMIC PROPERTIES

Metal matrix composites are under evaluation for use in rotating machinery such as jet turbine engines. The cyclic imposition of tensile, bending or combined loads creates the environment for fatigue failure. Likewise operational vibrations can strike resonant frequencies for structural parts. Thus the fatigue properties and damping capacity are of extreme importance to the utilization of such composites in high performance applications. Property characterizations have indicated that longer fatigue life at a higher stress level can be achieved with most reinforcements in metal matrices. And the combination of the elastic deformation of the reinforcing filament plus the plastic deformation of the matrix creates a hysterisis loop type of cyclic loading diagram which results in outstanding damping capacity.

### a. Fatigue

The interpretation of fatigue results on composite materials are severely complicated by the variety of loading schemes and definitions of fatigue failure. Each type of test defines a completely different type of stress cycle and stress field. The magnitude of the mean and alternating stresses, the strength and modulus of the filament and matrix, the surface sensitivity of the filament, the strength of the interfacial bond and the notch sensitivity of the filament and matrix are all involved in the type of fatigue curve that is recorded. Indeed the complexity of analysis of available fatigue data led Morris (125) to conclude that each composite system may behave uniquely, depending on the relative strength properties and characteristics of the components, mode of deformation and method of fabrication.

The characteristic form of a reverse bending fatigue diagram and the effect of temperature upon fatigue properties is illustrated in Figure  $52^{(53)}$ . A temperature increase to  $250^{\circ}$ F has little effect on the fatigue curve while at  $500^{\circ}$ F the curve is distinctly lower. Similarly combined axial/flexural fatigue testing at an A ratio of .95 (A= $\frac{Alternating\ Stress\ Mean\ Stress\ Vields\ a very flat fatigue\ curve\ but\ shows\ little\ effect\ of\ temperature\ to <math>500^{\circ}$ F, Figure  $53^{(49)}$ . The comparison with  $2024\ Al\ and\ Ti-6Al-4V\ alloys\ shows\ the\ distinct\ advantage\ of\ the\ composite\ relative\ to\ its\ likely\ competition\ in\ airframe\ or\ engine\ applications.$ 

In axial tensile fatigue both Kreider<sup>(39)</sup> and Schaefer<sup>(97)</sup>, Figure 54, have reported data on specimens of almost equivalent filament volume fractions but with significantly different initial strengths. However, the fatigue curves appear to merge at 10<sup>6</sup> cycles indicating that for fatigue limited applications tensile strength optimization may be of little benefit.

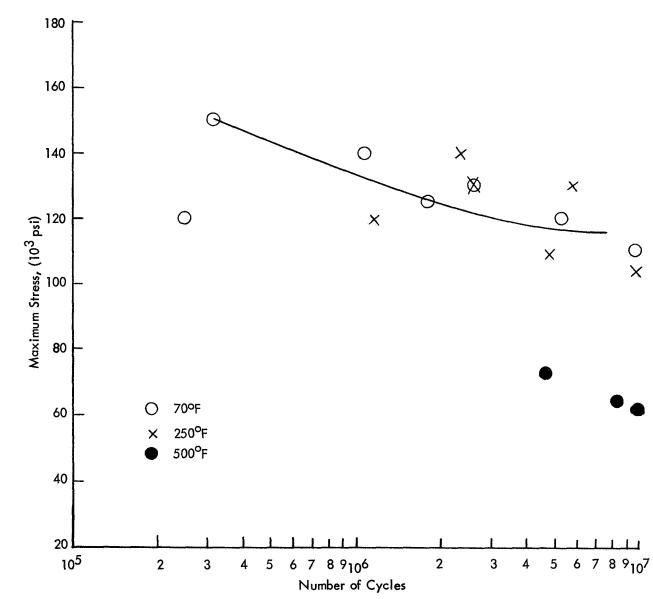


Figure 52. Al-42 v/o Boron Reverse Bending Flexural Fatigue Test Results for 0° Filament Orientation Specimens (53)

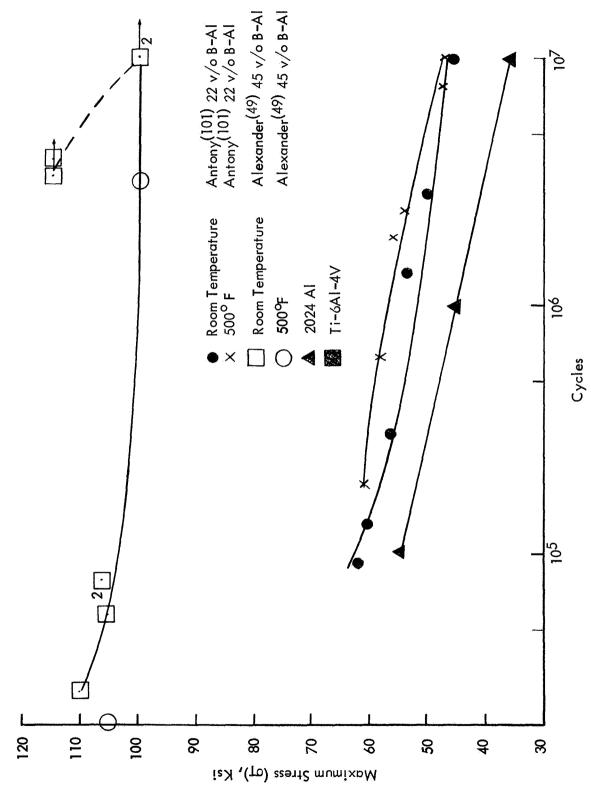


Figure 53. Combined Axial/Flexural Fatigue Results on AI-B Composites (A = 0.95)

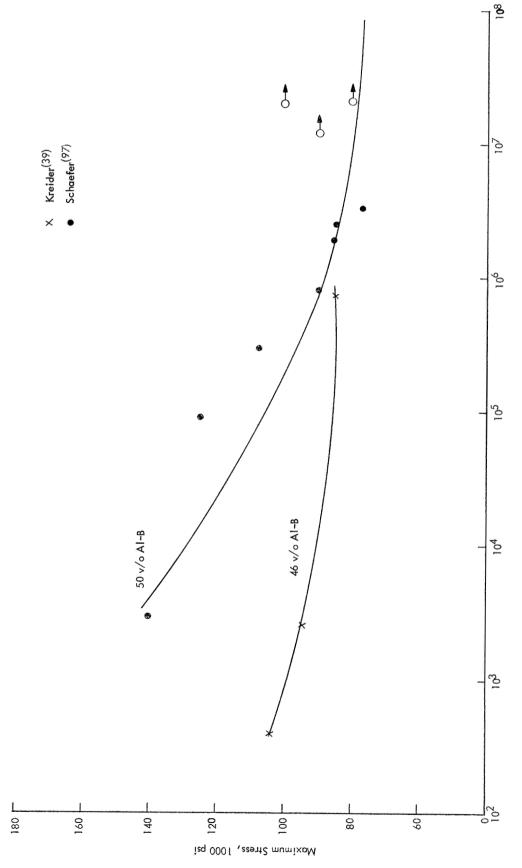


Figure 54. Axial Fatigue Results for Al-B Composites

Baskey  $^{(69)}$  examined the cyclic tensile behavior of nickel alloy matrix composites reinforced with tungsten and molybdenum wires. The improvement in fatigue performance at room temperature is illustrated in Figure 55. Again the characteristic flatness of the fatigue curve is clearly demonstrated. This effect is vividly demonstrated by the representation of the data at room and elevated temperature as a ratio of the stress to cause failure after  $10^6$  cycles divided by the tensile strength of the composite  $\sigma_{\rm c}$ , Table VI.

Table VI Comparison of  $\frac{\sigma_{cf}}{\sigma_{c}}$  for Hastelloy Composites and Matrix at Various Temperatures

Volume Percent Filament	℃f/ <sup>o</sup> c	Temperature
0	0.53	70 <sup>0</sup> F
37-W	0.61	70°F
36-TZM	0.70	70 <b>°</b> F
0	0.46	1500°F
23-W	0.90	1500 <sup>0</sup> F
0	0.25	1800 <sup>0</sup> F
30-W	0.67	1800°F

The incorporated filaments clearly improve the matrix fatigue properties at all temperatures but the improvement becomes more significant with increasing test temperature. The work of Dean  $^{(56)}$  was also conducted on the Ni-W system and demonstrated improvements in fatigue life at both room temperature and  $500^{\circ}$ C.

A series of papers (125,87,123,82,90,109) have been published which study the process of fatigue in filament reinforced composites as opposed to characterizing the fatigue properties of various systems. These papers collectively demonstrate the difficulties associated with the conduct of controlled scientific experimentation on complex phenomena occurring in composite materials. There is no unqualified picture of the process of fatigue in metal matrix composites. There is substantial information about the performance of a variety of materials, in a variety of conditions tested in a variety of ways. With those conditions Table VII is a compilation of fatigue "facts", a composite of the most generally recited observations from the detailed studies conducted to date.

Morris (125) conducted tensile fatigue tests on melt infiltrated Ag-W and Agsteel composites. The improvement of fatigue properties as a function of volume percent filament is illustrated in Figure 56. The pronounced benefit of continuous filament reinforcements is illustrated in Figure 57. The results of Ham (87) on embrittled W reinforced copper composites formed by liquid infiltration run counter to those of Baskey and Morris in that only relatively small increases in the stress to cause failure at  $10^{\circ}$  cycles was observed with increasing volume percent reinforcement and the ratio of  $\sigma_{\rm CF}/\sigma_{\rm C}$  decreased substantially with increasing filament volume percent. Clearly the brittle tungsten filament began to fracture after the first few cycles of test stress. The broken filaments initiated matrix fatigue fracture and the stress concentration caused by the matrix cracks was suffi-

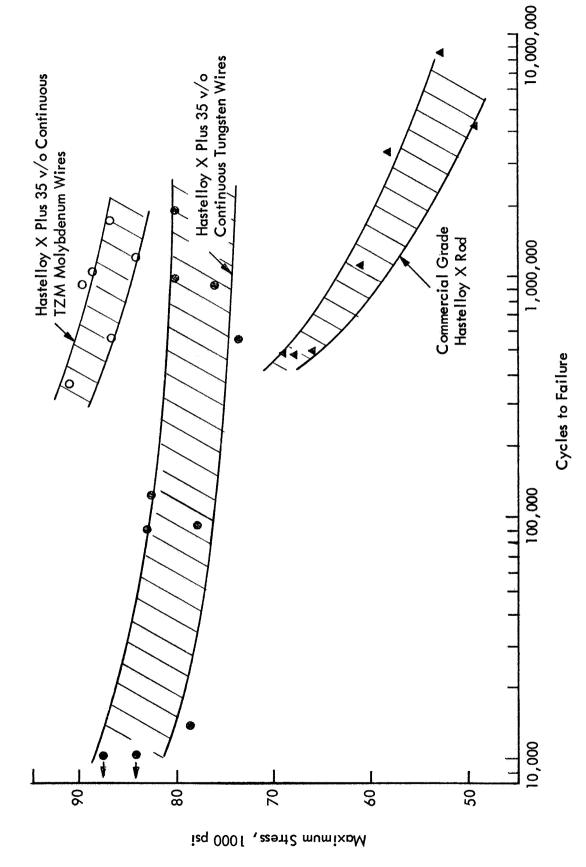


Figure 55. Direct Stress Fatigue Life of Hastelloy X Composites and Fiber-Free Hastelloy X at Room Temperature

Table VII Fatigue "Facts"

Fracture need not initiate at an external surface.

Work hardening can increase the likelihood of

fracture propagation thru a filament.

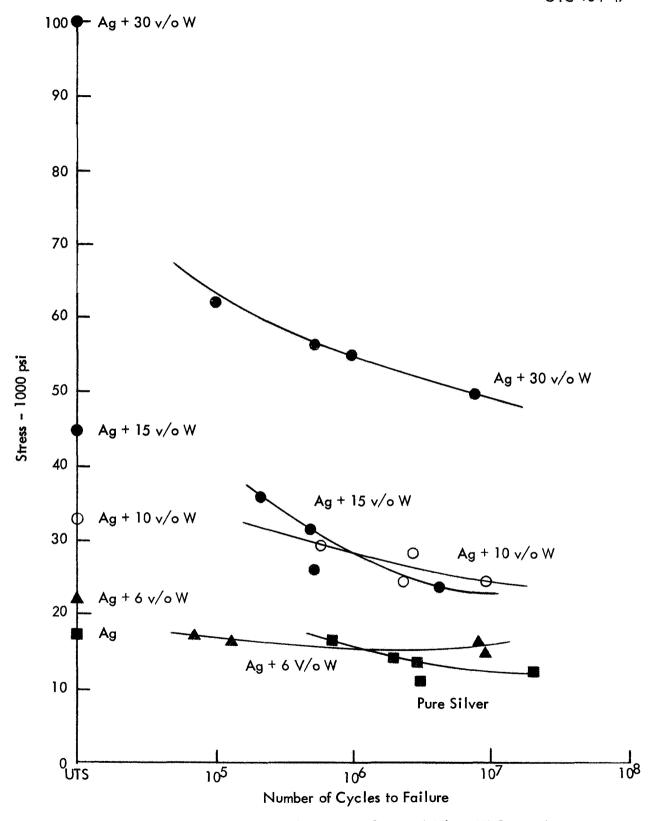


Figure 56. Fatigue Behavior of Silver and Silver-W Composites

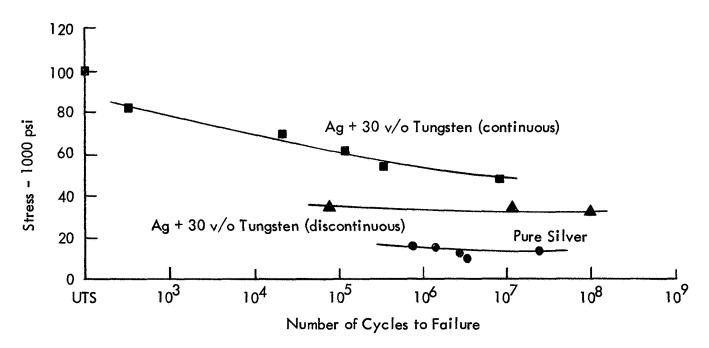


Figure 57. Comparison of Continuous and Discontinuous W Reinforced Silver Composites in Fatigue

cient to fracture subsequent filaments or sever the filament-matrix interface to the site of an adjacent filament fracture.

Baker (123,82,90,109) has conducted the most comprehensive study of fatigue behavior utilizing a reverse bending type of test on aluminum precoated SiO2 fiber and stainless steel wire reinforced hot-pressure bonded composite specimens. The SiO2 fiber has essentially the same modulus as the matrix, forms an excellent interfacial bond but is surface sensitive. The stainless steel wire has a modulus almost three times that of the matrix, develops a poorer but thermally variable interfacial bond strength, is insensitive to surface abrasion and is capable of plastic deformation. The reverse bending type of fatigue test imposes the complications of complete tensile-compressive cycling. This requires that an arbitrary level of loss in compressive properties be used as a measure of failure in the composite. Finally, the fatigue results are consistently presented as a function of the plastic strain range. The experimental results correlate well on this basis as should be expected since the measure of fatigue failure is loss in matrix integrity. This type of representation tends to mask the fact that composite fatigue life is considerably greater at higher stress levels for the composites and tends to overemphasize the fact that matrix integrity is lost when the matrix would be expected to fail according to the plastic strain range criteria of Coffin (124):

$$N_f^{1/2}$$
  $\Delta \varepsilon_p = c$ 

where

= number of cycles to failure

 $\Delta \varepsilon_p$  = the plastic strain range c = a constant = 1/2 the true fracture strain.

While the aluminum - 45 v/o SiO<sub>2</sub> and aluminum-stainless steel composites appear to follow the correlation between plastic strain range and fatigue life, Figure 58, the strong deviation exhibited for the aluminum - 25 v/o SiO<sub>2</sub> composites at fatigue lifes less than 2 x 10<sup>5</sup> cycles prohibits the broad generalization of this technique for predicting fatigue behavior in a wide variety of composite systems.

The effect of increased temperature on the fatigue life of aluminum-SiO<sub>2</sub> composites at various stress levels was observed to be slight, Figure 59, in agreement with the earlier observations on Al-boron composites. This is consistent with the fact that the plastic strain range would not be expected to increase greatly with increasing temperature relative to the increase in ductility of the matrix to fracture.

The type of investigations of the individual properties of composite materials which has begun to evolve for fatigue behavior are essential to the establishment of a base of knowledge beneath the superstructure which technological innovation and material characterization has constructed. The more advanced composite systems whether practical or model can now be fabricated with sufficient consistency to permit the resolution of the pertinent questions about composite behavior as opposed to observing the idiosyncrasies of the material on the basis of abbreviated data and discoursing at length.

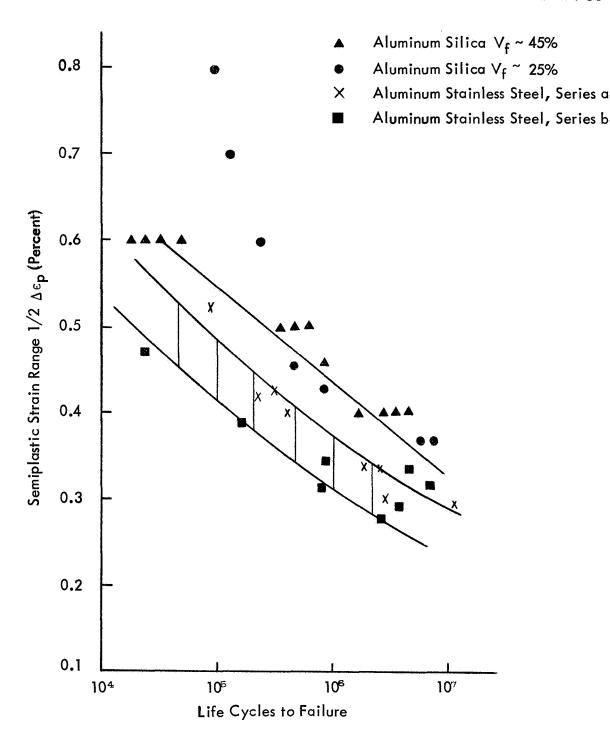


Figure 58. Semi-plastic-strain-range, calculated from the idealized model versus log cycles to failure for aluminum/silica and aluminum/stainless steel composites with  $5 \times 10^{-2}$  mm ( $2 \times 10^{-3}$  in.) diameter fibers.

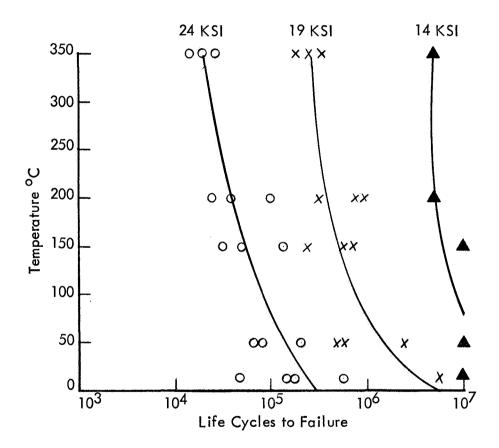


Figure 59. Temperature Versus Log Cycles to Failure for Aluminium/Silica Composites Tested Various Stresses

# b. Damping Capacity

The efficient utilization of the increased strength which is imparted to a composite by the incorporation of reinforcing fibers is dependent upon the deformation of the matrix into its plastic range. Thus a hysteresis loop type of stress-strain diagram, not unlike that for cast irons, is generated upon cyclic loading. This type of behavior is particularly effective under applicational conditions where resonant vibrations occur. The absorption of vibrational energy by the composite undergoing cyclic loading results in exceptionally good damping capacity as indicated in Figure 60 where the aluminum-SiO<sub>2</sub> composite is observed to exceed grey cast iron in damping capacity at higher stress levels.

### c. Impact Properties

The observed notch insensitivity, high strength, and the ability of the fibrous reinforcements to divert a propagating crack are materials characteristics which would be expected to yield improved impact properties. The data of Baskey on the nickel base composite systems reinforced with W and Mo indicate that the reinforcing fibers do indeed impart improvements in the impact energy absorption as compared with similarly processed matrix material at both room and elevated temperatures as high as 1600°F. However, the best composite system Hastelloy X-35 v/o TZM exhibited only 1/3 the impact energy adsorption of wrought Hastelloy X at room temperature and 1/2 at 1200°F. Thus the frame of reference becomes important. Scientifically the fiber reinforcement enhanced the mechanical properties of the matrix. Applicationally the impact strength of fiber reinforced Hastelloy X is inferior to the competitive form of that alloy. This is a relatively common situation in comparative illustrations of the potential of filament reinforced composites.

Kreider<sup>(39)</sup> has reported Charpy impact values of 2.5 ft. lbs. for half size specimen tests on 54 v/o BORSIC/aluminum composites. This relatively low energy absorption characteristic for brittle fiber reinforced composites was also demonstrated by Compton<sup>(96)</sup> where the room temperature energy absorption for A1-B and A1-SiC ranged from 5 to 10 in. lb., A1-Be was at 15 in. lbs., Ti-TZM at 25 in. lbs. and A1-stainless steel was 80 in.lbs. as compared to Ti-6A1-4V or stainless steel which absorbed in excess of 130 in.lbs.

The poor impact properties of such composites is reproduced in applicationally oriented ballistic impact tests where in terms of both visual evaluation of extent of damage and the reduction in strength of impacted specimens, the boron and silicon carbide reinforced aluminum composites are inferior to those with Be or stainless steel as a reinforcement. Impacts at velocities as low as 150 ft./sec. cause a strength degradation in the affected area of approximately 50% in boron reinforced aluminum specimens.

#### 4. MISCELLANEOUS PROPERTIES

The early composite fabrication programs concentrated on the simple tensile properties of generated samples and tensile property optimization was followed by a stage of surveying a broad selection materials properties. The general engineering importance of the

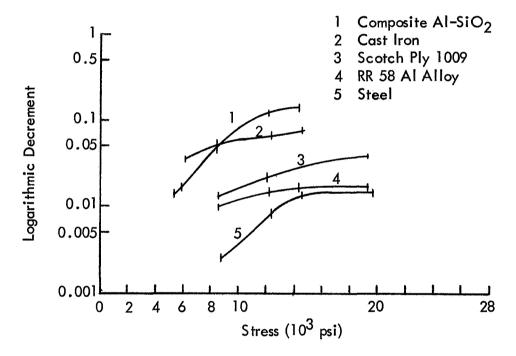


Figure 60. Comparison of Damping Capacity of Al–SiO<sub>2</sub> Composites with Conventional Engineering Materials

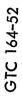
elevated temperature properties and of the dynamic properties has resulted in considerably more effort being concentrated on their detailed characterization especially in systems which offer the greatest potential for practical application. However, the scattered experimental results on composite compressive, flexural, shear and torsional properties will be important to the utilization of these materials in specific high performance applications. Similarly an understanding of the effects of corrosion and stress corrosion on the structural integrity of composite materials and of the ductility that can be expected from various types of composite becomes increasingly important as these materials advance toward actual use in aerospace components.

### a. Compressive Properties

The compressive strength of composite specimens has been observed to be approximately twice the tensile strength. Schuerch (26) reported compressive strengths of  $188 \times 10^3$  psi and  $344 \times 10^3$  psi for magnesium boron composites with 52 v/o and 71 v/o filament respectively. He presented a simplified model for composite compressive behavior based upon the shear crippling theory. His bend and compressive strength data are plotted in Figure 61 along with the more extensive compressive strength data of Alexander (25) as a function of volume percent filament and can be observed to scatter about the values predicted by the model. The character of the compressive failure was detailed by Alexander as shown in Figure 62. Figure 62a shows that the spreading of filaments can occur when the load application platen is harder than the reinforcing filaments. If the platen is softer than the filament (hardened steel), the ends of the specimen are embedded in the platen and failure occurs by progressive buckling of the boron filaments from the outer surface of the specimen inward, Figure 62b. The high compressive strength values of 236 x  $10^3$  psi and  $457 \times 10^3$  psi measured by Alexander at 47 v/o and 69 v/o B respectively were tested on steel platens while the lower values were measured on WC platens. Tensile specimens could not be tested to tensile failure because of grip slippage or shear but tensile loads as high as 200,000 psi were measured on the 69 v/o specimens. Longitudinal delamination can occur as a failure mode especially if the composite specimen is not uniformly and completely infiltrated, Figure 62. The compressive failure is localized to the region adjacent to the ends of the specimens in all well-fabricated composites and retests on the unaffected portion of tested rods yield equivalent compressive strengths.

The compressive strength of 22 v/o B-Aluminum rod composites as a function of temperature and as compared to the matrix alloy is demonstrated in Figure  $63^{(30)}$ . The tensile strength of these composites was 95,000 psi. Similarly, Kreider measured compressive strengths of 248,000 psi and 297,000 psi on rod composites which had tensile strengths of approximately 160,000 psi.

A more extensive series of tests conducted by Schaefer (97) on sheet material are compared in Table VIII. A strong specimen length effect was reported by Alexander (25) for 25 v/o B-Al hot-pressure bonded composites where 1/2-inch long specimens are tested at more than twice the strength of one inch specimens. Obviously the specimen configuration and test procedure has a strong effect in determining the value of compressive strength in metal matrix composites. However, the phenomenon of resistance to compressive loading involves much more than test procedure and should be examined in greater detail. The



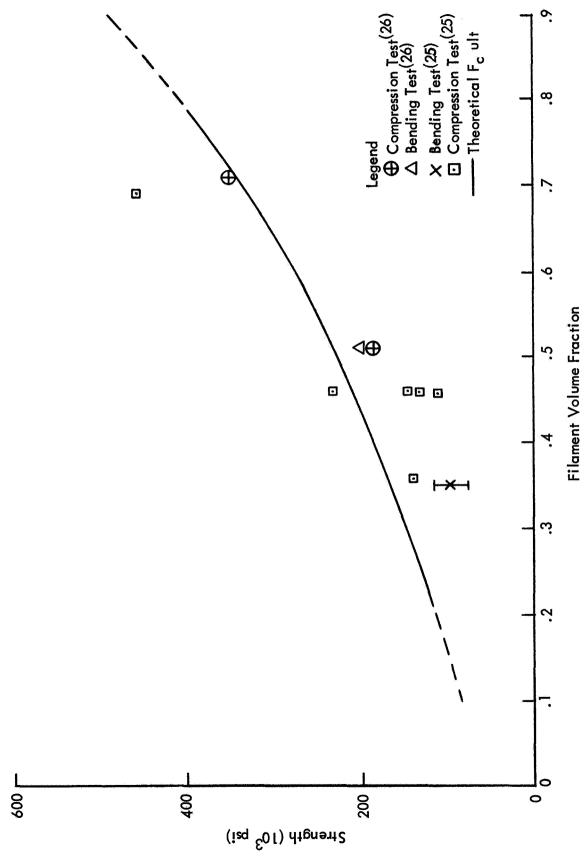


Figure 61. Predicted and Experimental Strength vs Packing Density for B/Mg Composites

# GTC 56-31

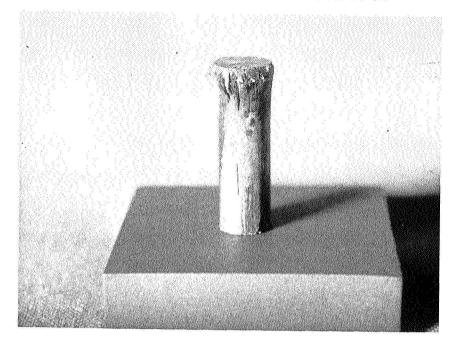


Figure 62a. Accordion End Failure in a Boron-Magnesium Compression Specimen

GTC 56-32

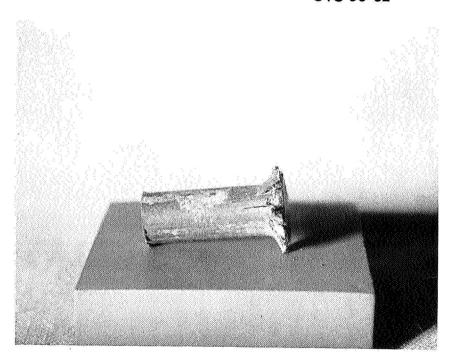


Figure 62b. Mushroomed End Failure in a Boron-Magnesium Compression Specimen

# GTC 56-33

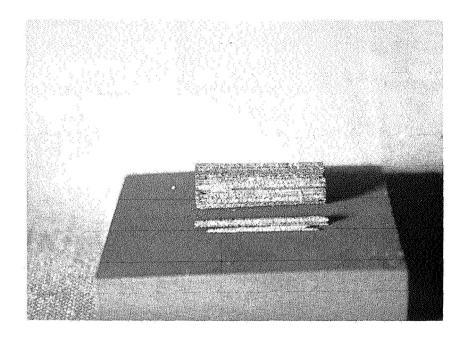


Figure 62c . Longitudinally Delaminated Boron-Magnesium Compression Specimen



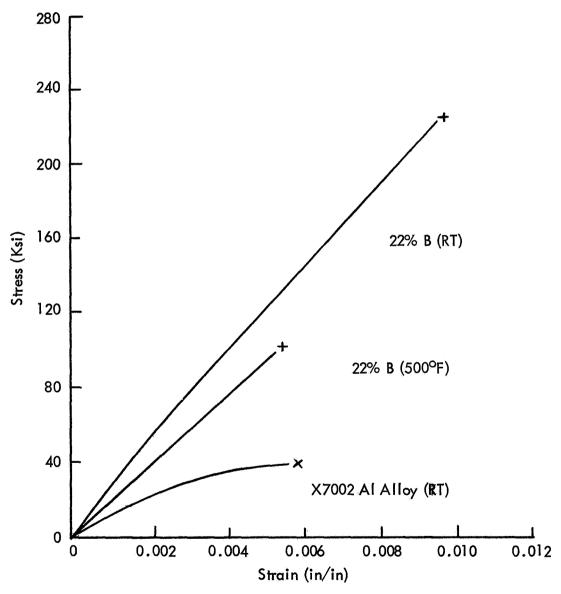


Figure 83. Compression Stress-Strain Curves of Unreinforced and B-Reinforced X7002 Al Alloy

effect of lateral constraint at specimen ends has been demonstrated but a composite with hoop oriented glass-resin reinforcement around an axial high volume percent boron-magnesium composite should be an attractive structural member for compressive load bearing capabilities. Similarly, investigations of the effect of modifying matrix shear properties, filament diameter or the interfacial bond strength should provide the basis for composite optimization for compressive applications.

Table VIII Comparison of Tensile and Compressive Strength of Unidirectional Aluminum Boron Composites

<u>v/o</u>	Tensile Strength (10 <sup>3</sup> psi)	Compressive Strength (10 <sup>3</sup> psi)	C.S./T.S.
50	175	164	1.06
37	121	156	1.26
25	82	122	1.48

### b. Flexure Strength

The measurement of flexure strength provides a type of test which eliminates the grip or load application problems which made tensile or compressive testing subject to criticism. The mechanical gripping of filaments in a wedge or compression grip may not provide data representative of the composite but rather may simply utilize the matrix as a mechanical load transfer medium. And the criticality of parallel and faces, accurately perpendicular to the specimen axis for compression testing is essential to the generation of meaningful data. But the simplicity of flexure testing and the added information regarding interfacial bonding which it can provide qualify it for serious consideration as production evaluation tool. The value of flexural strength calculated by simple beam theory is a measure of fabricational consistency but the character of the ultimate failure can be far more informative about the filament-matrix and matrix-matrix bonding accomplished in fabrication. As with reverse bending fatigue testing, flexure testing evaluates the specimen as a composite rather than a parallel array of filament and matrix.

Lenoe  $^{(111)}$  utilized two types of specimen for flexure testing, liquid aluminum infiltrated-65 v/o boron rods and hot pressed aluminum coupons containing 10 and 20 v/o boron, Figure 64. The hot-pressure bonded specimens exhibit flexure strength roughly double the tensile strength of the same type of specimen while the higher volume percent liquid infiltrated rod has flexural strength about 1.5 times its tensile strength.

The data of Alexander (25) for 35 v/o B-magnesium hot-pressure bonded composites indicate a similar 2X relationship between the flexure and tensile properties of that system, Table IX. A span-to-depth ratio range from 35:1 to 100:1 appears to have little influence on the flexure strength for this system which exhibits excellent filament-matrix bonding. Schuerch achieved a flexure strength value of 100,000 psi for a liquid infiltrated 52 v/o B-Mg composite.

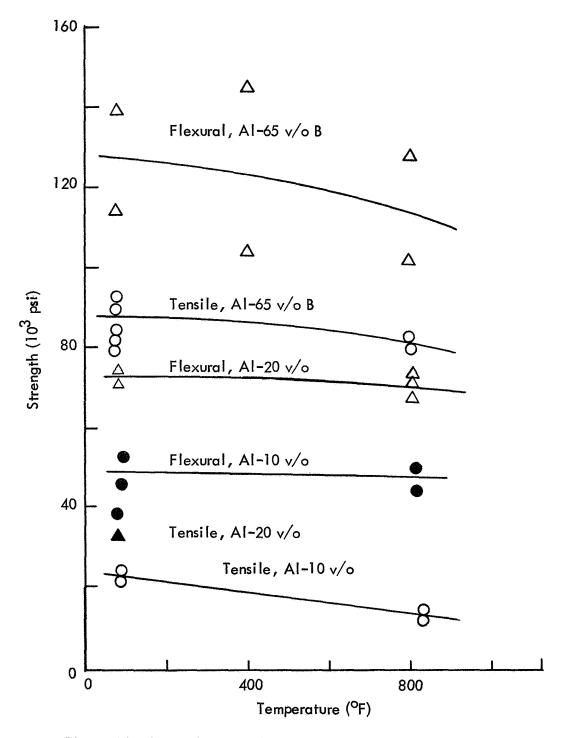


Figure 64. Strength Versus Temperature for Aluminum-Boron Composites

Table IX The Flexure Properties of 35 v/o B-Mg Composites with Various Span-to-Depth Ratios as Compared with Tensile Properties

Specimen	Type of	e bui	Strength	Modulus
Number	Test	Span: Depth	(× 10 <sup>3</sup> )	(x 10 <sup>6</sup> )
61	Tensile		53.9	26.5
62	Flexure	100:1	112	18.8
62A	Flexure	35:1	114	16.2
62B	Flexure	35:1	103	17.1
63	Flexure	<i>75</i> :1	98.4	16.4
63A	Flexure	35:1	97.5	1 <b>6.</b> 5
63B	Flexure	35:1	<i>7</i> 9.5	15.8
64	Flexure	75:1	116	18.1
64A	Flexure	35:1	11 <i>7</i>	16.9
64B	Flexure	35:1	104	16.6

The flexural strength test will be sensitive to both the extent of filament degradation and to the strength of the interfacial bond between filament and matrix. Since degradation can be independently monitored by matrix dissolution and filament testing this type of testing can serve as a ranking test for the propensity of consolidated filament and matrix to act as a composite.

# c. Torsional Properties

The torsional strength and modulus of axially aligned melt infiltrated 65 v/o B-Al composites has been measured by Lenoe (111) and the maximum shear strength in torsion for hot-pressure bonded 42 v/o B-Al by Young (53). The strengths are plotted in Figure 65. The magnitude of the shear stresses which can be sustained by such composites is quite impressive. With axially aligned solid rods only a fraction of the tensile strength of the filaments can be utilized in such a test. It would be interesting to evaluate  $45^{\circ}$  helically wound thin walled tubing as a potential light-weight unidirectional drive shaft material.

## d. Shear Properties

The shear strength of composite specimens has been measured by three different types of tests as depicted in Figure 66a. The double shear specimen was utilized by Schaefer (97) to document the effect of temperature on the shear strength of several different types of composite specimens, Figure 66b. The data of Kreider (39) using a single shear type of specimen at room temperature are in good agreement with the 50 v/o unidirectional material. Short shear beam evaluations of interlaminar shear have not yielded results which correlate well with the slotted specimens. There are two distinct shear stresses which are geometrically dissimilar and should be expected to yield different results, Figure 67. The inplane type of test is conducted with the interlaminar short shear beam type specimen and tests both the matrix-matrix and matrix-filament bonds in a hot-pressure bonded specimen. Short shear beam shear strengths would reflect differences in either

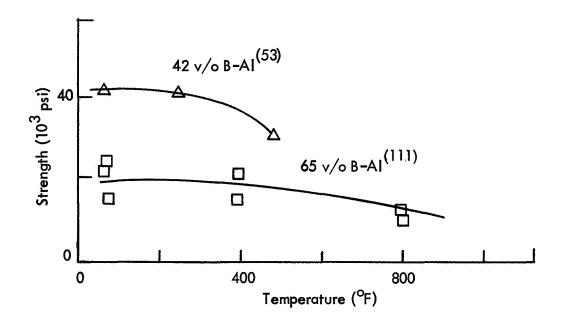


Figure 65. Maximum Shearing Stress in Torsion for AI-B Composites

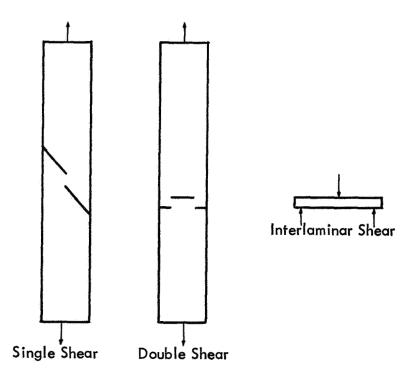


Figure 666. Types of Shear Test Specimens

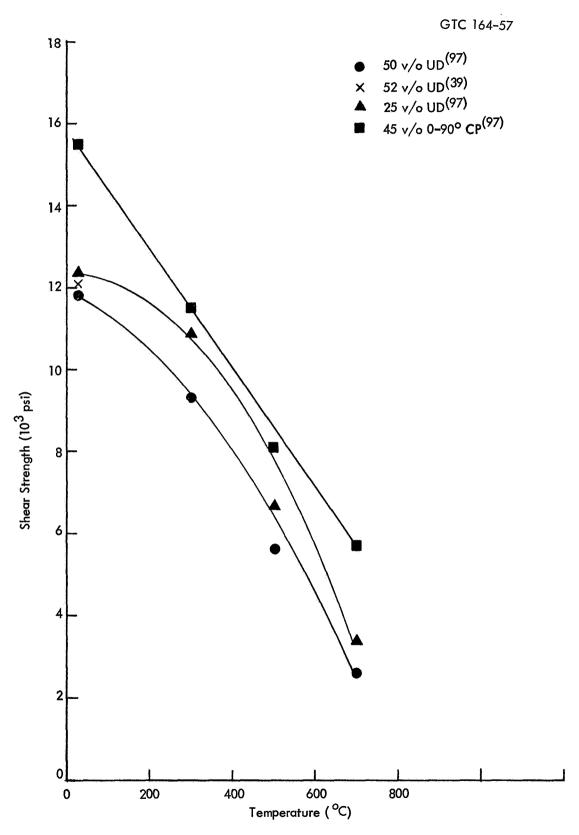


Figure 66b. The Effect of Temperature on Shear Strength for Unidirectional and Crossply Al-B Composites

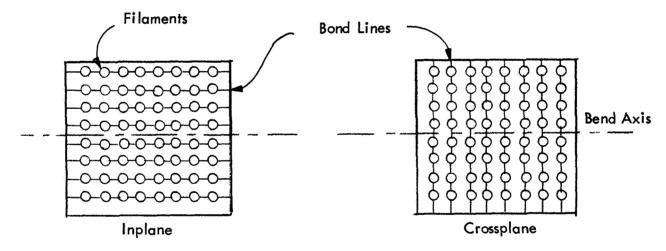


Figure 67. Geometrically Dissimilar Short Shear Beam Specimens

type of bond as a result of the fabrication history. The crossplane type of short shear beam test has not been reported but the single or double slit tensile specimens examines only the crossplane shear properties and thus does not reflect variations in matrix-matrix bond strengths.

Shear modulus data have been extensively evaluated by Young  $^{(53)}$  in the Al-B system as a function of filament orientation and temperature. Figure 68 presents the average test results for two different sizes of Al-42 v/o B panel tested in accordance with ASTM Specification D805. The shear modulus is observed to be significantly greater for all composite orientations except the 0-90° orthogonal specimens. The room temperature shear modulus data of Kreider ranged from 6.4 to 9.5 x  $10^6$  psi using two different test techniques and 38 and 52 v/o filament which shows relatively good correspondence considering the non-standardized test involved.

# e. Corrosion Properties

The effect of corrosive environments upon a variety of metal matrix composites was evaluated by Wolff<sup>(34)</sup>. Aluminum, copper and nickel matrix composites reinforced with boron exhibited weight changes which were, in general, equivalent to those of the matrix metals. Likewise the exposure of aluminum-boron composites to quite severe corrosive environments resulted in relatively modest degrees of degradation. Compton (96) confirmed that there were no gross differences in general corrosion of Al-stainless steel and Al-B composites. However, the inconsistencies in both works were attributed to variable degrees of exposure of filament. It is precisely the degree of attack down interfaces that is of interest regarding long-term application of composites in corrosive environments. A specimen design which might be useful in assessing the degree of attack in a quantitative fashion is pictured in Figure 69. A transverse tensile specimen from relatively thick composite plate with a reduced cross section which is narrow in the filament direction would be particularly sensitive corrosive attack down the filament-matrix interface since it maximizes the exposure of the filament ends and assures that small penetrations will yield measurable effects upon tensile strenath. The same type of specimen could be utilized for time delay failure evaluation of stress corrosion.

Kreider<sup>(39)</sup> exposed boron reinforced aluminum composites to stresses ranging from 22,500 psi to 60,000 psi for 17 to 37 days in salt spray with no evidence of either localized or general corrosive attack. No specimen failures occurred. Certainly more extensive studies are in order to assure that metal matrix composites can be confidently applied to components exposed to corrosive environments.

Schaefer (97) evaluated bare and coated composite specimens exposed to 5 percent salt spray for multi-hundred hour tests. Bare composite specimens and the matrix alloy were severely corroded by this type of test. Coated specimens provided varying degrees of protection with coating failures resulting in subsequent edge attack where boron filaments are exposed. A combination of analogization and epoxy polyamide primer treatment of specimen edges seemed to provide the best protection.

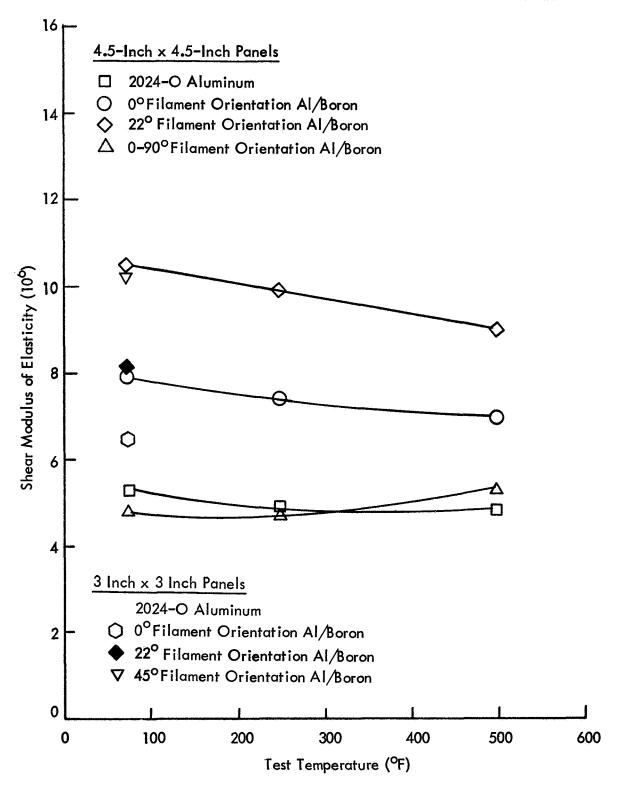


Figure 68. Average Shear Modulus Plate Test Results Vs Test Temperature for the Various Filament Orientation Al/Boron Panels and 2024-O Aluminum Sheet

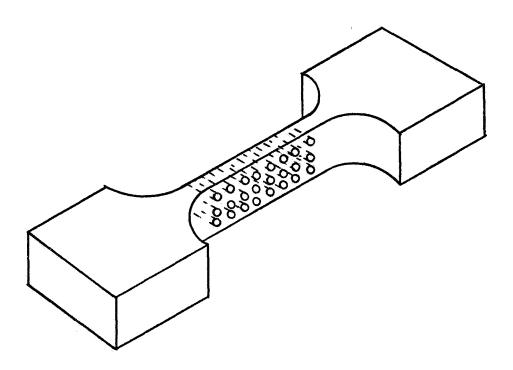


Figure 69. Environmental Test Specimen

### f. Joining Properties

The joining of composite materials is an area which has received only scattered survey evaluation. The subject has been only recently reviewed by Metzger (126). He concluded that "the joining of metal matrix fiber-reinforced materials has been neglected". His plea was that experimental investigations of joining methods should be initiated immediately rather than await the optimization of a particular system and find that application is inhibited by the lack of suitable joining procedures.

Kreider<sup>(39)</sup> has found that specimens hot pressure fabricated with interrupted layers in the reduced cross section failed at the layer interruption, Figure 70. These tensile specimens, with two of the six layers containing interruptions, exhibited two-thirds of the tensile strength of similarly fabricated continuous layer specimens.

An extensive series of evaluations are being completed by Schaefer (97). This work represents the most extensive joining and forming investigation yet conducted on advanced composites. Spot welds were generally able to support 400 to 600 pounds of load before failure and design loads of 25% and 40% of the unidirectional strength are recommended for spots on 1 inch centers for crossply and uniaxial material respectively. Resistance seam welds exhibited 40% to 45% point efficiencies calculated relative to the virgin composite strength. Riveted joints were so low as to result in design limits of 20% of the base composite strength

GTC 164-60

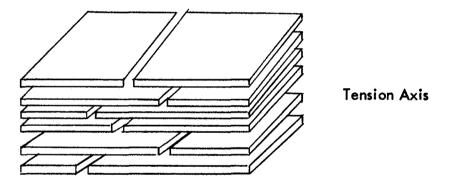


Figure 70. Schematic Drawing of Six Layer Bonded Composite with Seams Transverse to Gage Section

#### SECTION V

#### **PROBLEMS**

The potential of metal matrix composites as the structural material of the future has been experimentally demonstrated, but if that potential is to be realized the problems with these materials must be defined and attacked. Continuous filament reinforced metal matrix composites can be fabricated in a variety of forms, by a variety of techniques to yield structural elements with a broad range of impressive density-normalized mechanical properties. But the properties of the best composite systems in the simplest form are inconsistent and both the raw materials and fabrication processing are extremely costly. The lack of truly thermally stable low density filamentary reinforcements sets a practical limitation upon the temperature range which can be considered for the fabrication and application of metal matrix composites. The mechanical and some physical properties of composite materials have been measured but the basic understanding of the origins of those properties has not been developed.

The problems of consistency, cost and flexibility of form are inhibiting design utilization of metal matrix composites. The limited range of thermal stability is a second order design utilization problem but the availability of a more thermally stable filament specifically for metal matrix use would minimize this consideration.

The third order obstacles to the design utilization of metal matrix composites are really another category of problems, those relative to the understanding of their mechanical behavior. A considerable advantage of composites to the designer is the multiplicity of design trade-offs that a tailored material can offer. But those options can only be exercised if a complete understanding of the origins of the various types of behavior can be developed. This effort is tedious and can be likened to the experimental task which faced K. K. Kelly when he undertook to generate the thermodynamic tables which serve as the basis for the development of a science to replace the art of process metallurgy. Full acceptance of the art of composites will require the study of each of the various properties exhibited by composite materials in terms of their origins. The properties of the constituents, their modification and their interaction must be as thoroughly documented as the heats of formation of potential chemical reactants. Simple composite strength theory tells us that

$$1/2$$
  $\begin{bmatrix} Al (s) \\ 10,000 \text{ psi} \end{bmatrix} + 1/2 \begin{bmatrix} B (s) \\ 500,000 \text{ psi} \end{bmatrix}$  can equal  $\begin{bmatrix} Al-B \text{ (comp.)} \\ 255,000 \text{ psi} \end{bmatrix}$ 

but our understanding of the reaction route unlike the Kelly analogy is necessary to make the reaction go. The understanding of each of the properties exhibited by a composite will require the repetitive evaluation of the effect of a multitude of operating variables upon the constituents, their modification and their interaction. A composite is a system, and the number of operating variable requires that a systems approach be applied if the rapid exploitation of the art of composites is to be accomplished.

### 1. CONSISTENCY

The problem of consistency is of primary importance because a high performance material must be stretched to its ultimate in the provision of desired properties. If it is capable of attaining high performance but does so inconsistently it must be applied as a medium performance material. Consistency is also the number one problem because it is essential to have a material whose base condition can be defined if a basic understanding is to be derived as to the origins of its various properties. In Section III the blueprint for accomplishment of consistency in composite processing was described. Table III emphasizes that the statistical nature of composite failure must be acknowledged; the filament reinforcement must be represented in a fashion pertinent to its functioning in the composite to yield the desired property. The effect of processing variables or environmental conditions upon the reinforcement and its matrix must be monitored and the constituent characterization must be directly related to composite performance. The load translation process in a functioning composite can be understood in terms of the character and strength of the interfacial bond which is developed in fabrication and modified in service.

Table III indicates that the pieces of an experimental program for the improvement of consistency in metal matrix composites have been conducted. The collection of those pieces into a single program focused upon the following aspects of the fabrication procedure which have been observed to contribute to product variability will define the level of consistency which must be excepted from these materials.

- a. The variability of the filament
- b. The fabrication degradation
  - (1) Thermal degradation
  - (2) Environmental degradation
- c. The filament-matrix bond
- d. The matrix-matrix bond
- e. Thermal effects upon the matrix properties
- f. The residual stress distribution in the composite.

The question of cost and form quickly becomes involved when the decision is made to attack the problem of consistency. The work on the model system Cu-W and Al-SiO<sub>2</sub> has illustrated the value which can be gained in terms of understanding when a series of studies begin to generate interlocking pieces of information on specimens fabricated similarly. But the problem of consistency should be overcome in an applicationally viable system with a process capable of yielding the desired form of composite. Boron is the advanced filament which is the least costly in the U.S. and aluminum is the stable matrix that can be envisioned as having the largest potential for aircraft structure or engine blade application. Hot-pressure bonding is the experimental process which has been demonstrated to be capable of yielding useful forms of these materials. Thus Al-B is a composite system which has been identified as the "stalking horse" system. The concentration on the Al-B composite system has resulted in significant improvements in product consistency on the basis of production experience. Davis<sup>(31)</sup> has reported that coefficients of variation

have progressed downward from 36.4 percent in July of 1967 to 26.3 percent in March of 1968. Coefficients of variation on more recently generated panels which have been exposed to a selection process which eliminates poorly consolidated panels by nondestructive test techniques have been lowered to between 10 and 15 percent. Such product improvement is a tribute to the influence of standardized procedures and extended production experience. However, each size scale-up results in a reversion in product consistency. Indeed Davis indicates that "an extended detailed study will be necessary in order to establish just what parameters are most important in controlling and eliminating this variation in properties".

### 2. COST

Hot-pressure bonding is a batch process as currently practiced and has exhibited both scale-up problems and fabrication costs which run three to four times the cost of the incorporated filaments. It yields a form of material which must compete with biaxial alloys. Hot-pressure bonding has been carried to the most advanced state of development in applications programs for aircraft structural elements as a result of the joint effort between General Dynamics Convair and the Harvey Aluminum Co. An analysis of costs for hot-pressure bonding which has evolved from this fabrication program has indicated that the price for 50 v/o Al-B composite in multi-ton quantities would be \$500-\$600 per pound. A range which clearly defines the problem of cost. The manual labor, the number of process steps, the batch processing, the capital investment and the power requirements of the hot-pressure bonding process as a source of large dimension stock for aerospace structural use may impose a severe limitation on the applicability of this new form of material. It may be more competitive when evaluated relative to the direct production of an end item such as a jet engine blade. In either case the reduction of reinforcement cost, the automation of the filament handling and precursor preparation processes, and the minimization of scrap generation and thermal exposure times are the task areas where concerted effort can define the route to more economical metal matrix composites. The step hot pressing os large composite panels with small dies is an attempt to overcome the problems of direct scale-up to large panel size pressing capabilities. And the continuous hot-pressure bonding process of Schmitz $^{(42)}$  for laboratory scale tape preform production is a conceptually sound approach to the potential reduction of composite processing costs. Plasma sprayed preforms in themselves are currently costly but the preform route to metal matrix composite production may provide cost savings in subsequent steps by alleviating the need for protective atmospheres or organic binder exhaustion systems while accommodating filament orientation tasks.

But ultimately cost reduction is a task for those involved in competition for a market. And a real and definable market is essential to the generation of cost competition. Since metal matrix composites are not in use and the present market is only evaluational it should not be surprising that great strides have not been made in metal matrix composite costs. But as the properties become more fully documented and the potential applicational utility becomes more specifically defined, the routes to composite fabrication must be increasingly scrutinized with regard to volume producibility.

The need for a market to motivate cost consciousness might be satisfied by accentuating the utility of the state of the art composites and composite forms. The design engineer can assist in the short-time scale transition of composites from laboratory to assembly line if he will accept the material for what it is today and consciously seek uses for it. Likewise, those involved in composites production need to assess the portfolio of fabrication processes and concentrate on those which show inherent potential for low cost production. In that vein the continuous casting process has three strong attributes and one distinct limitation. It is a continuous process which need add little processing cost to the cost of the incorporated filament, it should yield a consistent product since the thermal exposure cycle is subject to precise control, and it yields a uniaxial form of composite which can maximize the property advantage of the uniaxial composite over competitive conventional alloys. Its disadvantage is the necessity that the incorporated filament be stable relative to the liquid matrix at least for the time required to accomplish a solidification cycle. This disadvantage underlines the need for work on the development of filament reinforcements for metal matrix application. Filament reinforcements that can facilitate the task of reducing composite cost by permitting the exercise of fabricational processes which are inherently low cost in nature are needed.

### 3. FORM

The problems of composite form, flexibility of form and scale-up in size are similar in character to those of cost. Composites can be fabricated by a wide variety of processes in a wide variety of forms but the flexibility which is provided involves the utilization of widely differing technologies. Flexibility of form and size within each fabrication process is currently quite limited. Three types of composite forms seem most likely for process optimization; monolayer tape or precursor broadgoods, sheet or rod mill products, and high volume end items formed firectly from the constituents or a precursor.

The formation of tape or broadgoods is relatively simple and the immediate objective should be optimization of the various available processes to minimize degradation, accomplish consistent control of volume percent filament and filament spacing, and provide a compositing precursor which simplifies the subsequent consolidation into a finished composite form, all at minimal cost.

Composite processing to yield a sheet form mill product will have to provide for the accomplishment of consolidation with minimal filament degradation over a wide range of volume percent loading with any specified schedule of ply orientations in any specified thickness and should provide for the direct formation of any degree of single or double curvature. The scale-up of the hot-pressure bonding process to sizes which would be feasible for skin applications in aircraft or space vehicles involves the problems of providing uniform heating over a large surface area while applying a relatively high pressure at a reasonably elevated temperature (900°F) while maintaining a protective atmosphere and controlling the position of incorporated filament. The composite mill product is likely to be tailored not only in ply orientation and thickness to the ultimate application, but cut outs and contoured fittings may have to be fabricated into the sheet rather than being formed simple sheet stock. The extent to which cost reduction can be accomplished will dictate the degree to which wastage can be tolerated in subsequent assembly processes.

The rod form of composite has greater potential for mill product status of a conventional sort. The continuous casting process should be adapted to yield I-beam, hat section, Z-beam and tubular cross sections for evaluation as direct replacement high performance uniaxial materials. Process optimization will permit automated control over filament feed and the ability to form a variety of cross-sectional sizes and shapes from a single casting stand.

# 4. FILAMENT

Boron, silicon carbide coated boron, nitrided boron and silicon carbide filament together with beryllium, stainless steel, tungsten and molybdenum wire constitute the available continuous reinforcements for metal matrix composites. Concentrated effort on the reduction of cost and improvement in quality has advanced boron from a 300,000 psi filament at \$3000 per pound to a 500,000 psi filament at \$250 per pound. In spite of the highly reactive nature of boron relative to potential metallic matrices, the mechanical properties reported in Section IV indicate that boron-aluminum and boron magnesium composites are serious competitors for high performance applications. The lesser reactivity of silicon carbide or coated borons provides a greater latitude in time-temperature exposures but adds no higher temperature matrices for compositing consideration.

The effort currently being conducted on Al<sub>2</sub>O<sub>3</sub> filaments grown from the melt is the first filament development program directed specifically toward the generation of a fiber which will permit greater fabricational and applicational flexibility in terms of the time-temperature tolerance of the combination of non-equilibrium phases. In addition to Al<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub> has shown stability in Ti to 1100°C while TiC and B<sub>4</sub>C have exhibited stability in Ni to 1100° and 1000°C, respectively<sup>(38)</sup>. The limited number of available filaments for metal matrix compositing use is a problem. It limits the breadth of scope of the metal matrix composites field. It is important to recognize that progress in resin matrix technology has been greatly enhanced by concentration upon filaments which were specifically suited to resin matrix use. It is essential to the broadening of metal matrix composites technology that reinforcements be chosen for their stability in a metal rather than as an evaluational spinoff of resin matrix reinforcement efforts.

### 5. BEHAVIOR

The problems of composite behavior are problems of understanding. These are the problems of constructing and testing models for the behavior of metal matrix composites. They transcend the task of rationalizing the results of a few scattered tests against the postulations which have been presented to represent composite behavior. They must answer the questions:

- a. What is the fracture mechanism of a metal matrix composite?
- b. Can it be represented statistically?
- c. How is the character and strength of the interfacial bond influential?
- d. What is the load transfer mechanism at the interface?
- e. Can the character of filament, matrix and interfacial bond be used to interpret composite behavior?

- f. Can the models for the various types of behavior be made more definitive?
- g. How can the models best be tested in terms of the measurable characteristics of the composite?

These are basic questions and progress will be measured by the degree to which individual investigators ask these questions relative to sharply focused research programs in each of the property areas discussed in Section IV. None of the areas of metal matrix composite behavior have been thoroughly explored. The materials have been characterized. The focus on the Al-B system and the optimization of the hot-pressure bonding fabrication process for sheet material provides the basis for basic research efforts on a potentially useful system. The collection of data on the Cu-W and Al-SiO2 model system has underlined the value of multiple types of investigations on the same material. Using the most consistent available evaluational materials, the construction of a basic understanding of the behavior of metal matrix composites in each of the property areas can be undertaken. By concentrating on the best available composite material and defining a sharply focused set of research objectives relative to a specific type of behavior, vague and repetitious studies which evolve from the broader scope property evaluations can be eliminated. Definitive answers to small questions are apt to be cumulatively more informative than the multiple superficial treatment of entire areas of composite behavior.

How can the interfacial shear strength be evaluated? How can the interfacial tensile strength be evaluated? How do individual filament fractures accumulate to cause composite failure? Can filament degradation be identified nondestructively? Can residual stress or Poisson's ratio difference triaxial stress influences on composite strength be demonstrated? What is responsible for the off-axis property differences between Al-B and Al-Be composites? Can matrix strength influences on composite strength be delineated? Does the composite actually transmit the implied load to the filament or is the wedge type gripping simply applying load directly to the filaments via compression of the matrix? These are but a few of the small questions which evolve from the need to understand the tensile behavior of metal matrix composites. However, the complexity of the material, the number of parameters which must be monitored and the need for a statistically valid sample of each parameter makes the answer to a small question a large experimental task.

A similar series of questions have been developed in the discussion of individual properties in Section IV and will not be reiterated here. The influence of matrix properties, interfacial bonding, and filament notch sensitivity seem most important to fatigue studies. The magnitude of their importance can expect to vary with the type of fatigue test. Filament creep properties seem to determine composite creep behavior but the effect of filament fractures and the load redistribution process which occurs with progressive filament fracturing needs study as does the really long-term degradation in filament properties at potential application temperatures. The triaxiality imposed by Poisson's ratio differences between filament and matrix should be particularly noticeable with ductile filament reinforcements. The influence of matrix properties on the compressive strength and modulus of filament reinforced materials deserves experimentation.

Additionally, the accumulation of a collection of engineering design data for the most advanced composite system need not await the generation of a complete understanding of the origins of the respective properties. The work of Baskey<sup>(69)</sup> on Ni-W composites illustrates the consistent results which can evolve from a standardized fabrication procedure. Thus the establishment of a statistically viable representation of the obtainable properties of Al-B sheet composites will permit serious consideration of the materials for weight critical applications at an early date.

The basis for the research and development conducted on metal matrix composites to date, has been the magnitude of jump in mechanical properties which such materials potentially offered. The underlying assumption has been that "If a substance is created with properties an order of magnitude beyond the present art, there is bound to be someone who needs this". The experimental effort reported here, demonstrates that the predicted potential for metal matrix composites can be attained for a variety of properties. Continued progress in the optimization and understanding of these properties can be expected, however, the major contribution to composites technology is now in the hands of design engineers with a real need for the magnitude of jump which has been provided.

#### SECTION VI

#### CONCLUSIONS

- 1. Filament reinforced metals are anisotropic by nature and this characteristic makes it difficult to form direct and simple comparisons to the conventional state-of-the-art homogeneous metals.
- 2. The reinforcement of metals with high modulus filaments is a relatively recent development but the expected increase in unidirectional mechanical properties has been demonstrated with the most highly developed materials combinations.
- 3. Metal matrix composites have demonstrated outstanding density-normalized directional strength and modulus properties as well as improvements in the important engineering properties of fatigue, stress rupture, and creep. They are relatively notch insensitive with an excellent damping capacity.
- 4. While a relatively large number of reinforced metal-filament combinations have been investigated for engineering applications as well as model systems, the most significant advancements have been made with aluminum reinforced with boron, silicon carbide-coated boron, and beryllium and with magnesium reinforced with boron.
- 5. Many fabrication processes such as hot-pressure bonding, liquid metal infiltration or casting, electrodeposition, plasma spraying chemical vapor deposition, extrusion and rolling and high energy rate forming have been investigated to produce metal matrix composites. Only hot-pressure bonding and casting have evolved as practical processes to produce a usable composite structure. Plasma spraying and the deposition process are applicable to producing a preform for subsequent hot-pressure bonding. All fabrication processes produce fairly poor consistency in composites produced.
- 6. The economics of the fabrication process have been a deterrent to wide scale early utilization of metal matrix composites. It has been predicted that large hot-pressure bonded 50 v/o Al-B sheet will cost \$500-\$600/lb. in multi-ton quantities based on a \$250/lb. filament cost. Casting offers a potentially lower cost process for fabrication of a variety of unidirectional components or preforms for pressure bonding in liquid metal-filament stable systems such as magnesium-boron or aluminum-coated boron or silicon carbide.
- 7. Most property data on metal matrix composites have been generated on uniaxial material. The tensile strength and modulus for non-interacting systems that have been well characterized approximate law of mixture predictions regardless of the fabrication process. As an example, an aluminum 40 v/o boron composite utilizing 400,000 psi boron filament and 2024 annealed aluminum would have an approximate room temperature tensile strength of 177,000 psi and a modulus of 28 x 10° psi. The short time elevated temperature properties of Al-B begin to drop off between 500° and 600°F but maintain about 50% of the virgin strength to 1000°F. From very sparse data it appears

- that long time exposure to 500 hours at temperatures to 200°C in the Al-B system shows significant strength degradation while Al-BORSIC shows no effects to 1000 hours at 500°C.
- 8. The off-axis tensile properties of systems that are not well bonded such as Al-B or SiC asymptotically approach a value of about 20,000 psi at about 20<sup>o</sup> from the normal while well bonded systems such as Al-Be or Mg-B show good transverse properties approximating that of the matrix.
- 9. No apparent strain rate effect has been observed in metal matrix composites involving high modulus filaments. The notch sensitivity of all composite systems are as expected, very good, There were no notch effects with a stress concentration factor as low as 2.9 in Al-B composites. Poisson's ratio for Al-B and Al-SiO<sub>2</sub> composites have been measured in the range of .2 to .374 on uniaxial and crossplys.
- 10. The creep properties of composites are controlled primarily by the creep rates of the incorporated filament. In the case of Al-B composites creep rates in the range of  $10^{-5}$  to  $10^{-4}$  in/in/hr at temperatures to  $1200^{\circ}$ F have been observed.
- 11. The stress rupture properties of composites are excellent and are controlled by the load carrying capacity of the reinforcing filaments. The stress rupture life of Al-B systems have shown virtually a straight line plot vs temperature to 1000 hours whereas comparative homogeneous aerospace materials fall off quite rapidly. For comparison 50 v/o BORSIC-Al has a stress rupture life of 70,000 psi at 500°C for 1000 hours compared to 20,000 psi for Ti6Al-4V at the same conditions.
- 12. Miscellaneous properties such as compressive, flexure, torsional, shear, etc. are very sparse and require specific interpretation based on matrix-filament combinations, fabrication history and test method.
- 13. The dynamic properties of metal matrix composites are generally quite good but their interpretation are complicated by the variety of loading methods creating a vastly different stress field and cycle. Generally the fatigue strength increases with increasing volume percent of filament while fracture does not necessarily initiate at an external surface. In a combined axial/flexural fatigue test the run out (10<sup>7</sup> cycles) stress for 45 v/o B-Al composite at room temperature was 115,000 psi and 1000,000 psi at 500°F compared at room temperature to 38,000 psi for 2024 Al and 90,000 for Ti-6Al-4V.
- 14. The impact properties of composites seem to be governed by the type of reinforcing filament and the bonding between the matrix and filament. Systems such as Al-B have poor impact properties, while A-Be and Al-stainless steel have progressively better impact properties although still less than 1/2 as good as Ti-6Al-4V.
- 15. There is no currently available stable filament for titanium and superalloy class matrices. Coated boron and SiC all react to yield composites of only limited value. Experimentally TiC, TiB<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> have been shown to be stable for these important aerospace matrix materials.

#### SECTION VII

#### RECOMMENDATIONS

- 1. Continuing research and development efforts should be directed toward the accomplishment of improvements in the cost, consistency or degree of confidence with which metal matrix composites can be recommended for high performance application.
- 2. Fabrication development efforts should concentrate on inherently low cost processes which yield broadly useful composite forms.
- 3. More emphasis should be placed on the development of preforms, such as tapes or sheets, that are low cost and easily processed into final structural form.
- 4. The provision of incentives for the design utilization of composites should be considered as a means of creating an early market for the currently available simple forms of filament reinforced materials. An artificially induced market would provide both cost consciousness on the part of potential producers and progressively increasing confidence on the part of designers.
- 5. While volume production and process automation can be expected to improve product consistency, emphasis should be placed on the generation of a basic understanding of the interactions (physical, chemical and mechanical) between filament and matrix during fabrication and in use.
- 6. Confidence in the use of metal matrix composites can be enhanced by the generation of a comprehensive picture of the origins of the impressive creep, fatigue, stress rupture, impact resistance, damping capacity and notch insensitivity of this new material.
- 7. More specifically, detailed characterization of the boron-aluminum or BORSIC-aluminum systems should be conducted to fully define the engineering properties of the most advanced system to permit maximum consideration of applications potential.
- 8. Efforts should be initiated to develop new filamentary reinforcements fully stable at processing and operating temperatures. Growing requirements are for filaments compatible with titanium and superalloys.
- 9. Above all else the utility of filament reinforced composites is dependent upon the field maturing rapidly. The focus of the next generation of experimental accomplishments must be on the problems of metal matrix composites rather than the potential of metal matrix composites. The realization of the demonstrated potential is dependent upon problem acknowledgement, definition and solution.

#### REFERENCES

- Kelly, A. and Davies, G. J., "The Principles of the Fibre Reinforcement of Metals", Metallurgical Reviews, 10, 37, 1–77 (1965).
- Cratchley, D., "Experimental Aspects of Fibre-Reinforced Metals", Metallurgical Reviews, 10, 37, 79-144 (1965).
- Jech, R. W., McDanels, D. L. and Weeton, J. W., "Fiber Reinforced Metallic Composites", Proc. 6th Sagamore Ordnance Materials Research Conference, Composite Materials and Composite Structures, 1959 (AD 161 443), pp. 116–139.
- 4. Machlin, E. S., "Status Report on Non-Metallic Fibrous Reinforced Metal Composites", Materials Research Corp., Contract No. NOw 61-0209-c (AD 265 943), U. S. Navy, Sept. 1961.
- 5. Baskey, R. H., "Fiber Reinforcement of Metallic and Non-Metallic Composites: Phase I State of Art and Bibliography of Fiber Metallurgy", Clevite Corp., ASD Interim Report 7-924(I), U.S. Air Force Contract No. AF 33(615)-7139, Feb. 1962.
- 6. <u>Fiber Composite Materials</u>, Papers presented at a Seminar of the American Society for Metals, Oct. 1964, American Society for Metals, Metals Park, Ohio, 1965.
- 7. Fiber Strengthened Metallic Composites, ASTM STP 427, 1967.
- 8. Metal Matrix Composites, ASTM STP 438, 1968.
- 9. Krock, R. H., Broutman, L. J. (Edt.), <u>Modern Composite Materials</u>, Addison Westley Publishing Co., 1967.
- 10. Holister and Thomas, Fiber Reinforced Materials, Elsevier Publishing Co., Ltd., Amsterdam, London, N. Y., 1966.
- 11. Composite Materials, Inst. of Metallurgists, American Elsevier Publishing Co.
- 12. Kelly, A., "Fiber Reinforcement", Strong Solids, Oxford, 1966, p. 121.
- 13. Strengthening Mechanisms Metals and Ceramics, Syracuse V. Press.
- 14. Zackay, V. F. (Edt.), High Strength Materials, John Wiley & Sons, Inc. 1965.
- 15. Advanced Fibrous Reinforced Composites, Society of Aerospace Material and Process Engineers, Western Periodicals Co., N. Hollywood, California, Vol. 10, 1966.
- 16. Advances in Structural Composites, Society of Aerospace Material and Process Engineers, Western Periodicals Co., N. Hollywood, California, Vol. 12, 1967.

- Advanced Techniques for Material Investigation and Fabrication, Society of Aerospace Material and Process Engineers, Western Periodicals Company, N. Hollywood, California, Vol. 14, 1968.
- 18. 14th Refractory Composites Meeting, Air Force Materials Laboratory, Dayton, Ohio, AFML-TR-68-129, August 1968.
- 19. Jackson, C. M. and Wagner, H. S., Fiber Reinforced Metal-Matrix Composites: Government Sponsored Research, DMIC Report 214, August 28, 1967.
- 20. Hanby, K. R., Fiber Reinforced Metal-Matrix Composites, 1967, DMIC Report S-21, June 1, 1968.
- 21. Rauch, H. W., Sutton, W. H., McCreight, L. R., Survey of Ceramic Fibers and Fibrous Composite Materials, AFML-TR-66-365, October 1966.
- 22. Stowba, A., Fiber Metallurgy, A.S.M. Bibliography Series B79-14, 1965 Sept. 1967 (inclusive).
- 23. Metal Matrix Composites, Defense Documentation Center Report Bibliography Search Control No. A 96463.
- 24. Thomas, Huffadine, and Moore, "Preparation, Properties, and Application of Metal/Ceramic Mixtures", Mets Reviews, Vol. 8, No. 32, 1963, p. 461.
- 25. Alexander, J. A., Withers, J. C. and Macklin, B. A., <u>Investigation of Three Classes of Composite Materials for Space Vehicle Application</u>, NASA CR-785, May 1967.
- 26. Schuerch, H., Compressive Strength of Boron-Metal Composites, NASA CR-202, April 1965.
- 27. Petrasek, D. W., Elevated-Temperature Tensile Properties of Alloyed Tungsten Fiber Composites, NASA TN-D-3073.
- 28. Kelly, A. and Tyson, W. R., <u>J. Mech. & Phys. of Solids</u>, <u>13</u>, 1965, p. 329.
- 29. Ahmad, I. and Barranco, J. M., Strengthening of Copper with Continuous Tantalum Filaments, Watervliet Arsenal Report 6835, October 1968.
- 30. Toy, A., Atteridge, G. D., and Sinizer, D. I., <u>Development and Evaluation of the Diffusion Bonding Process</u>, AFML-TR-66-350, November 1966.
- 31. Davis, L. W., "Aluminum-Boron Composites", 14th Refractory Composites Meeting, AFML-TR-68-129, August 1968, p. 201.

- 32. Kreider, K. G. and Leverant, G. R., Boron Fiber Metal Matrix Composites by Plasma Spraying, AFML-TR-66-219, July 1966.
- 33. Cunningham, A. L. and Alexander, J. A., "The Fabrication, Evaluation, and Mechanical Properties of Aluminum Matrix Composites", contained in <u>Advances</u> in <u>Structural Composites</u>, Society of Aerospace Material and Process Engineers, Western Periodicals Co., North Hollywood, California, Vol. 12, October 1967, p. AC-15.
- 34. Wolff, E. G. and Hill, R. J., Research on Boron Filament/Metal Matrix Composite Materials, AFML-TR-67-140, June 1967.
- 35. Hoffmanner, A. L., Study of Methods to Produce Composite Beryllium Blades, Final Report, Contract NOw-65-0281-f, August 12, 1966.
- 36. Herman, M., Beryllium Wire Metal Matrix Composites Program, Final Report, Contract N 00019-67-C-0532, Allison Report EDR 5950, July 31, 1968.
- 37. Sumner, E. V., <u>Development of Ultrahigh Strength Low Density Al Plate Composites</u>, Final Report, NAS8-11508, Harvey Report No. HA-2263, July 1966.
- 38. Alexander, J. A., Cunningham, A. L. and Chuang, K. C., <u>Investigation to Produce Metal Matrix Composites with High Modulus</u>, Low Density Continuous Filament Reinforcements, AFML-TR-67-391, February 1968.
- 39. Kreider, K. G., Schile, R. D., Breinan, E. M. and Marciano, M., <u>Plasma</u>
  Sprayed Metal Matrix Fiber Reinforced Composites, AFML-TR-68-119, July 1968.
- 40. Cunningham, A. L., Davies, L. G. and Alexander, J., "The Characteristics of the Mg-B Composite System", contained in <u>Advanced Techniques for Material Investigation and Fabrication</u>, Society of Aerospace Material and Process Engineers, Western Periodicals Company, North Hollywood, California, Vol. 14, November 1968, p. II-1B-2.
- 41. Herman, M., R & D of an Advanced Composite Technology Base and Component Fabrication Program for the Turbine Compressor and Fan Blades, Final Report, AF 33(615)-5321, Allison Report EDR-5332.
- 42. Schmitz, G. K. and Metcalfe, A. G., <u>Development of Continuous Filament Reinforced Metal Tape</u>, AFML-TR-68-41, February 1968.
- 43. Glasser, J. and Sump, C. H., <u>Evaluation of Manufacturing Methods for Fibrous Composite Materials</u>, AFML-TR-65-175.
- 44. Sutton, W., "Fiber Reinforced Metals", Modern Composite Materials, Edts. Broutman-Kroch, Addison Wesley, 1967, p. 412.

- 45. Harman, C. G., Non-Glassy Inorganic Fibers and Composites, NASA SP-5055, August 1966.
- 46. Davis, L. W., "How Metal Matrix Composites are Made", Fiber-Strengthened Metallic Composites, ASTM STP 427, 1967, p. 69.
- 47. Thornton, H. R., "Fabrication of Metal Matrix Composite Materials", <u>Jnl. of Composite Materials</u>, II Vol., January 1968, p. 32.
- 48. Herzog, J. A., Filaments, Fibers and Metal Matrix Composites, AFML-TR-67-244, October 1967.
- 49. Alexander, J. A., Cunningham, A. L. and Withers, J. C., "Techniques for the Fabrication of High-Strength, High-Modulus, Low-Density Filament-Reinforced Metal-Matrix Composites", Materials Technology An Interamerican Approach, The American Society of Mechanical Engineers, N. Y., N. Y., May 1968, p. 301.
- 50. Alexander, J. A., "Five Ways to Fabricate Metal Matrix Composite Parts", Materials Engineering, July 1968, p. 58.
- 51. Alexander, J. A. and Withers, J. C., "Procedures of Fabrication and Properties of Ceramic Filament-Reinforced Metals", <u>Summary of the 14th Refractory Composites Working Group Meeting</u>, AFML-TR-68-129, August 1968, p. 229.
- 52. Snide, J. A., Lynch, C. T. and Whipple, L. D., Current Developments in Fiber-Reinforced Composites, AFML-TR-67-359, February 1968.
- 53. Young, J. H., Advanced Composite Material Structural Hardware Development and Test Program, Preliminary Results from Air Force, Contract F 33615-68-C-1156.
- 54. Stuhrke, W. F., "The Mechanical Behavior of Aluminum-Boron Composite Materials", Metal Matrix Composites, ASTM STP 438, 1968, p. 108.
- 55. Shimizu, H., Doloway, J. F. and Taylor, R. J., Mechanical Behavior of Al-B Composites, Preliminary Results from Air Force Contract AF 33615-68-C-1165.
- 56. Dean, A. V., "Reinforcement of Ni-Base Alloys with High Strength Tungsten Wires", National Gas Turbine Establishment Report NGTER-266, April 1965 (AD 464 771).
- 57. Michalik, S. J. and Weinstein, M., Silicon Carbide Filaments, preliminary data on Contract N 00019-68-C-0177.
- 58. Alexander, J. A. and Davies, L. G., "Continuous Casting as a Composite Fabrication Process", 15th National SAMPE, Western Periodicals, North Hollywood, Calif., Vol. 15, April 1969.

- 59. Alexander, J. A., Shaver, R. G. and Withers, J. C., A Study of Low Density High Strength, High Modulus Filaments and Composites, NASA CR-523, July 1966.
- 60. Ahmad, I., Creco, V. P., and Barranco, J. M., Electroforming of the Composites of Nickel Reinforced with Some High-Strength Filaments, Watervliet Arsenal Technical Report WVT-6709, January 1967.
- 61. Adsit, N.R., "Electroforming to Make Composite Materials", Fiber Strengthened Metallic Composites, ASTM STP 427, October 1967, p. 27.
- 62. Withers, J. C. and Abrams, E. F., "The Electroforming of Composites", <u>Plating</u>, June 1968, pp. 605–611.
- 63. Baker, A. A., Harris, J. T. and Holms, E., "Fiber-Reinforcement of Metals and a Filament Winding & Electroforming Technique", <u>Metals & Materials</u>, Vol. 1967, p. 211.
- 64. Bonnano, F. R. and Withers, J. C., "The Fabrication and Mechanical Properties of SiC Filament Metal Matrix Composites", contained in Advanced Fibrous Reinforced Composites, Society of Aerospace Material and Process Engineers, Western Periodicals Co., North Hollywood, Calif., Vol. 10, November 1966, p. F-105.
- 65. Abrams, E. F. and Shaver, R. G., Filament-Metal Matrix Composite Motor Case Study, Final Technical Report, Contract NOw-66-0616-c, August 1967.
- 66. Greening, T. A., Advanced Wire-Wound Tungsten Nozzles, AFPL-TR-67-181, April 1967.
- 67. Withers, J. C., The Fabricatility of Multilayer Beryllium Wire Reinforced Aluminum Composites by Chemical Vapor Deposition, GTC Tech. Report 155.5-1, July 1968.
- 68. Greszczuk, L. B., "Filament Reinforced Refractory Metals", contained in Advanced Fibrous Reinforced Composites, Society of Aerospace Material and Process Engineers, Western Periodicals Co., North Hollywood, California, Vol. 10, November 1966, p. F-15.
- 69. Baskey, R. H., <u>Fiber-Reinforced Metallic Composite Materials</u>, AFML-TR-67-196, September 1967.
- 70. Adamski, R., Bkattachargga, S. and Parikh, N. M., Influence of Fabrication Variables on the Properties of Fiber-Reinforced Metals, IITRF Report B6074-6, Final Report, Contract N 00019-67-C-0372.
- 71. Meyerer, W. J., The Feasibility of Forming a Boron Fiber Reinforced Aluminum Composite by a Hot Extrusion Process, AFML-TR-68-127, August 1968.

- 72. Robinson, R. K. and Farnsworth, P. L., "Tungsten Fiber Reinforced Nickel Composites", 10th Refractory Composites Working Group, AFML-TR-65-207.
- 73. Sump, K. R. and Robinson, R. K., "Fabrication of Refractory Composites by Pneumatic Impaction", 11th Refractory Composites Working Group, AFML-TR-66-179, July 1966.
- 74. Robinson, R. K., "High Energy Rate Forming of Fibrous Composites", Fiber-Strengthened Metallic Composites, ASTM STP 427, 1967, p. 107.
- 75. Miller, G. E., Snajdr, E. A., Williford Jr., J. F., Investigation of Fiber Reinforced Metal Matrix Composites Using a High Energy Rate Forming Method, preliminary results on Contract F 33615-67-C-1422.
- 76. Snajdr, E. A., Williford Jr., J. F., "Investigation of Fiber-Reinforced Metal Matrix Composites Using a High Energy Rate Forming Method", 14th Refractory Composites Working Group, AFML-TR-68-129.
- 77. Rosen, B. W., "The Strength and Stiffness of Fibrous Composites", Modern Composite Materials, Addison Wesley, 1967, p. 114.
- 78. Grinius, V. G., Micromechanics Failure Mechanism Studies, AFML-TR-66-177, August 1966.
- 79. Lenoe, E. M., Micromechanics of High Strength, Low Density Boron Filament Reinforced Al Metallic Composites, Part I, AFML-TR-67-125, May 1967.
- 80. Conliffe, C. H., Advanced Composite Material in Turbo Machinery Components, AFML-TR-68-366, February 1969, Vol. 1 & II.
- 81. Joseph, E., "Processing Parameters for Diffusion-Bonded Al-Boron Filament Composites", thesis Air Force Inst. of Technology, GAW-B/Mich 67-9.
- 82. Baker, A. A., Mason, J. E., and Cratchley, D., "High-Strain Fatigue Studies of Composite Material", J. Mat. Sci., 1, No. 3, Aug. 1966, p. 229.
- 83. Toy, A., "Mechanical Properties of Be Filament Reinforced Aluminum Composites", J. of Materials, 3, No. 1, March 1968, p. 43.
- 84. Jackson, P. W., Baker, A. A., Cratchley, D., Walker, P. J., "The Fabrication of Components from Al Reinforced with SiO<sub>2</sub> Fibers", <u>Powder Metallurgy</u>, Vol. II, No. 21, 1968, pp. 1–22.
- 85. Cooper, G.A., "Orientation Effects in Fibre Reinforced Metals", <u>Jnl. of Mech.</u> & Phys. of Solids, 14 (2), 1966, p. 103.

- 86. O'Brien, D. J., Martin, P. W., and Williams, R. V., "Fiber Reinforced Metals by Electroforming", Applied Matls. Res., 5, 1966, p. 241.
- 87. Ham, R. K., and Place, T. A., "The Failure of Cu-W Fibre Composites in Repeated Tension", Jnl. Mech. & Physics Solids, 14 (5), 1966, p. 271.
- 88. Ellison, E. G. and Harris, B., "The Elevated Temperature Properties of a Ni Alloy Reinforced with W Wires", Applied Matls. Res., 5, 1966, p. 33.
- 89. Baker, A. A. and Cratchley, D., "Stress Strain Behavior and Toughness of a Fibre-Reinforced Metal", Applied Materials Res., 5, 1966, p. 92.
- 90. Baker, A. A., "The Effect of Fibre Volume Fraction and Interfacial Bond on the Fatigue of Al Reinforced with S.S. Wires", Applied Matls. Res., 5, No. 3, p. 143.
- 91. Cratchley, D. and Baker, A. A., "The Tensile Strength of Silicon Fiber Reinforced Al Alloy", Metallurgia, 69, 1964, p. 153.
- 92. Cooper, G. A., "Electroformed Composite Materials", <u>Jnl. of Matls. Science</u>, 2, No. 5, Sept. 1967, p. 409.
- 93. Weeton, John W., "Fiber Metal Matrix Composites", Machine Design, 41, No. 4, February 20, 1969, p. 142.
- 94. Cheskis, H. P., and Heckel, R. W., "In Situ Measurements of Deformation Behavior of Individual Phases in Composites by X-Ray Diffraction", Metal Matrix Composites, ASTM STP 438, Aug. 1968, p. 76.
- 95. Cheskis, H. P., Hattangadi, A. and Heckel, R. W., "In Situ Measurements of the Stress-Strain Behavior of Fiber and Matrix Phases in Metal-Matrix Composite Materials", 14th Refractory Composites Meeting, AFML-TR-68-129, Aug. 1968.
- 96. Compton, W. A., Stewart, K. P. and Monew, H., Composite Materials for Turbine Compressors, AFML-TR-68-31, June 1968.
- 97. Schaefer, W. H. and Christian, J. L., "Evaluation of the Structural Behavior of Filament Reinforced Metal Matrix Composites, Preliminary Data on AF Contract F 33615-67-C-1548.
- 98. Sumner, E. V., "The Effect of Residual Stress and Cold Work on the Load Deformation Curve of Aluminum Matrix Steel Fiber Composites", Advanced Fibrous Reinforced Composites, SAMPE, Vol. 10, Western Periodicals, North Hollywood, Calif., p.:F-11.
- 99. Sumner, E. V., "The Effect of Heat Treatment on the Physical Properties of Some Boron-Aluminum Alloy Matrix Composites", Advances in Structural Composites, SAMPE, Vol. 12, Western Periodicals, North Hollywood, Calif., p. AC-19.

- Taylor, R. S., Doloway, J. W. and Shimizu, H., Application of Composite Materials to Ramjet Inlet Structures, AFML-TR-68-85, May 1968.
- 101. Antony, K. C. and Chang, W. H., "Mechanical Properties of Al-B Composites", Transactions Quarterly, ASM 61, No. 3, Sept. 1968, p. 550.
- 102. Goodwin, V. L. and Herman, M., "Be Wire-Metal Matrix Composites Program", Preliminary Data on Contract No. N 00019-69-C-0234, Feb. 1969.
- 103. Carlson, R. G. and Tomalin, D. S., "Metal Laminate Composite Materials for Compressor Blading", <u>Advanced Fibrous Reinforced Composites</u>, <u>SAMPE</u>, Vol. 12, Western Periodicals, North Hollywood, Calif., p. F-45.
- 104. McDanels, D. L., Signorelli, R. A. and Weeton, J. W., Analysis of Stress-Rupture and Creep Properties of W Fiber Reinforced Copper Composites, NASA-TN D-4173, 1967.
- de Silva, T., "A Theoretical Analysis of Creep in Fibre Reinforced Composites", Jnl. Mech. & Phys. of Solids, 16, No. 3, 1968, p. 169.
- 106. Stowell, E. Z. and Liu, T. S., "On Mechanical Behavior of Fiber Reinforced Crystalline Materials", J. Mechanics & Physics of Solids, Vol. 9, 1961, p. 242.
- 107. Tsai, S. W., Strength Characteristics of Composite Materials, NASA Report CR 224, April 1965.
- 108. Jaclson, P. W. and Cratchley, D., "The Effect of Fibre Orientation on Tensile Strength of Fibre Reinforced Metals", <u>Jnl. Mech. & Phys. of Solids</u>, <u>14</u> (1), 1966, p. 49.
- 109. Baker, A. A., "The Fatigue of Fiber Reinforced Aluminum", <u>Jnl of Matls. Sci.</u>, 3, No. 4, July 1968, p. 412.
- 110. Baker, A. A. and Jackson, P. W., "Silica Fibre Reinforcement of Aluminum", Glass Technology, 9, No. 2, April 1968, p. 36-41.
- 111. Lence, E. M., Murro, R. P., Beaumont, S. and War, J., Micromechanics of High Strength, Low Density Boron Filament Reinforced Metallic Composites, AFML-TR-67-125, Part II.
- 112. Tsai, S. W., Structural Behavior of Composite Materials, NASA CR-71, July 1964.
- 113. Haskins, Z. and Rosen, W. B., "The Elastic Moduli of Fiber-Reinforced Materials", Jnl. of Applied Mechanics, Jan. 1964, p. 223.
- 114. Ellison, E. G. and Boone, D. H., "Some Mechanical Properties of Boron-Tungsten Boride Filaments", Jnl. Less Common Metals, 13, 1967, p. 103.

- Jones, R. C., "Deformation of Wire Reinforced Metal Matrix Composites", Metal Matrix Composites, ASTM STP 438, Am. Soc. for Testing & Materials, 1968, p. 183.
- 116. Kelly, A. and Tyson, W. R., "Tensile Properties of Fibre Reinforced Metals, II. Creep of Silver-Tungsten", J. Mech. & Phys. Solids, 14, 1966, p. 177.
- 117. Miura, I. and Honma, H., "On the Strength of W-Fiber Reinforced Al Composites at High Temperatures", <u>Jnl. Japan Inst. of Metals</u>, 31 (5), 1967, pp. 607.
- 118. Miura, I. and Honma, H., "On the Strength of W Fibre Reinforced Al Composites at High Temperatures", Jnl. Japan Inst. of Metals, 31 (5), 1967, pp. 607-610.
- Petrasek, D. W., Signorelli, R. A. and Weeton, J. W., Refractory-Metal Fiber Nickel-Base-Alloy Composites for Use at High Temperatures, NASA TN D-4787, Sept. 1968.
- 120. Alexander, J. A., Elevated Temperature Reactivity of Boron-Metal Matrix Composite Materials, AFML-TR-67-101, June 1967.
- 121. Alexander, J. A. and Stuhrke, W. F., "Fiber Strengthened Metallic Composites",

  The Effect of Temperature Upon the Mechanical Properties of Nickel-Boron Composites, ASTM STP 427, Oct. 1967.
- 122. Alexander, J. A., Stuhrke, W. F. and Chuang, K. C., "The Elevated Temperature Reactivity in Boron-Metal Matrix Composite Materials", <u>Advanced Fibrous</u>
  Reinforced Composites, SAMPE, Vol. 10, Nov. 1966, p. F-91.
- 123. Baker, A. A. and Cratchley, D., "Metallographic Observation on the Behavior of Silica Reinforced Alumina Under Fatigue Loading", Applied Mtls. Res., Oct. 1964, p. 215.
- 124. Tavernelli, J. F. and Coffin, L. F., "A Compilation and Interpretation of Cyclic Strain Fatigue Tests on Metals", Trans. Amer. Soc. for Metals, 51, 1959, p. 438.
- 125. Morris, A. W. H. and Steigerwald, E. A., "An Investigation of the Fatigue Behavior of Tungsten-Reinforced and Steel Reinforced Silver Composites", <u>Trans.</u> <u>AIME</u>, Vol. 239, 1967, p. 730.
- 126. Metzger, G. E., Welding of Metal-Matrix Fiber-Reinforced Materials, AFML TR-68-101.